ELEMENTAL ANALYSIS OF SOME GEOLOGICAL SAMPLES USING NEUTRON ACTIVATION TECHNIQUE

By

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The undersigned hereby certify that they have read and recommend to the Faculty of Agriculture and Environmental Science, Graduate Studies for acceptance a thesis entitled "**Elemental Analysis of Some Geological Samples Using Neutron Activation Technique**" by **Asres Yihunie Hibstie** in partial fulfillment of the requirements for the degree of **Doctor of Philosophy in Environmental Science**.

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Signature of Author

To my late, beloved, mother Etenesh Bogale Cherinet

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Abstract

This study describes the technique and procedures used for the simultaneous determination of elements involved in Instrumental Neutron Activation Analysis, applied to some geological samples as a sensitive and non−destructive tool. Using Isotopic Neutron Source, the delayed gamma−rays emitted due to neutron capture have been applied for the investigation of the elemental constituents and concentrations of major, minor and trace elements in such samples.

The experimental investigations were carried out at the scale of researcher's practices at the chosen site by measuring soil/rock nutrient contents from hydrologically isolated farmland/site plots. Using detailed sampling procedures with special precautions, against contaminations, to yield meaningful results, soil/rock samples have been collected from selected areas of East Gojjam, pulverized, homogenized and prepared for irradiation.

The samples were properly prepared together with known standards of potassium iodide or cupric sulphate or arsenic trioxide (one /or both at a time), and simultaneously irradiated in to isotopic neutron flux in the same irradiation position. After activation, the samples were subjected to gamma−ray spectrometry, using a high−purity germanium detection system and computerized multichannel analyzer. The gamma−ray spectra from the irradiated samples were measured by high resolution gamma−ray spectrometry with a calibrated germanium detector. Both qualitative and quantitative approach to data collection and analysis were used in order to gain insight into what should be acceptable to the community.

The results obtained have a practical interest in view of possible applications to the technology of extraction and exploration of a variety of industrial, metallic, cement minerals, and geothermal energy and coal deposits. These results tend to agree with the lowest values reported in the literature, and the analytical precision is sufficient for the determination of the standard deviation of the distribution of all elements in East Gojjam and these results were briefly discussed within the text of this research work.

In carrying out the analysis, the best and most convenient method being the Instrumental and /non−destructive Neutron Activation Analysis was adopted, and NAA has matured and attained analytical competence providing not only rapid, quantitative, qualitative, simultaneous, multi−element analysis but also crucial validation support to programmes using different analytical techniques. This method of analysis is generally multi−element and experimental conditions can be designed to be non−destructive to the sample, and it plays a leading role among analytical techniques in the field of environmental research related to human, animal and plant health.

Studies of different radionuclide and trace elements in the environmental samples are very important for health physics, research and education. Many forms of elements are possible in environmental samples which may be hazardous for human being, animals and also plants. Most of them have the potential for both beneficial and harmful effects.

Key words: Geological Samples, East Gojjam, INAA, extraction of minerals, multi−element analysis, radioisotopes, radiation exposure

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Yebrage Hawariat, East Gojjam, Ethiopia — Asres Yihunie Hibstie
June, 2012

Chapter 1

Introduction:

This Chapter introduces the problem surrounding this research endeavor. It also provides a rationale for the study and explains the research questions.

1.1 Background/Problem statement:

The problem was identified over the past 30 years, while living among the society/community lived in “Yebrage”, having smallholder farming plots and systems, in “East Gojjam” Administrative Zone, Ethiopia. Ethiopia is located in the north−eastern part of Africa (Reimann et al., 2003; Seleshi et al., 2003), neighboring some African countries, the horn of Africa and it was also called “bread−basket”.

Agriculture is the main economic activity practiced in “East Gojjam” and its surroundings. In the traditional, unmechanized, backward and rain−fed agriculture, a variety of grains, seeds, serials, crops (but not cash crops) and vegetables were grown. Crops produced here are not mostly high valued vegetables, fruits and beans.

The permanent residents kept limited number of domestic animals (goats, sheep, donkeys, horses, chickens and cattle). Animals grazed (Seleshi et al., 2003) first on the natural pasture around the village, and then moved into the tradition and rain−fed farms to feed on crops residues.

Land degradation is a major environmental/agricultural problem in East Gojjam. This marginal area is prone to declining yields (Gruhn et al., 2000) after a few years of cultivation because of their fragile soils and a thin nutrient base. After a few years of cultivations, the fields become abandoned, rejuvenated using flowering ("Gibto" (Lupinus Albus), beans, peas, etc.) plants per every "5" years, used organic manure (natural fertilizer) to renew their farm land, and the farmers moved to new areas.

The problem is further compounded by the low quantity and quality (low nitrogen N, phosphorus P and potassium K) and locally−derived fertilizers (cattle manure, crop residues, termitaria soil and leaf litter) used by smallholder farmers to supplement inorganic fertilizers. Such agricultural practices have resulted in abolishment of the traditional grazing, woodlot keeping and traditional farming pattern which assumed sustained production and development.

Inappropriate land use, poor management and lack of inputs have led to a decline in productivity, soil erosion, salination, and loss of vegetation (Baiono et al., 2006). The unplanned land uses could rely on such rotational use of land for centuries, producing wood, farm products and opening grazing land for their livestock.

Worsening poverty is closely linked to low and/or declining levels of agricultural productivity and increasing food insecurity. Agriculture underpins most rural livelihoods and national economies. In the medium term, enhancing agricultural growth and productivity are keys to a viable and widely applicable poverty reduction strategy. This has led to renew interest by development agencies in increasing agricultural productivity.

Already vulnerable to highly variable climatic conditions accelerating climatic change poses a further significant challenge to improving agricultural productivity in "East Gojjam" (Devereux & Edward, 2004). The rural livelihoods context, including climate related trends and shocks, together with people's capital asset bases varies over time and space resulting in a wide range of coping and adapting strategies.

A key challenge for local and national, particularly public sector, decision makers and politicians is to understand the context and strategies of farmers and other "stakeholder" in agriculture for coping and adapting the variable climatic conditions in order to engender innovation. However, for various reasons many of these decision makers seldom access or appear to be influenced by information in this form.

This research aims to facilitate a process of interaction and learning where information/knowledge from different sources (local, regional, national, and international) is shared and integrated in a way that results in its novel use by "stakeholder" in agricultural innovation systems to better adapt and variability. It will nominate to build on trans−disciplinary partnerships and initiatives in agriculture and natural resources and also to build on "East Gojjam" (regional adaptation programs of action), which are linked to external funds, and prioritize agriculture, including incremental changes in coping systems but also to differing extents of irrigation; and on farmers' coping/livelihood strategies; and zonal/district−based agricultural "stakeholder" (public and private) strategies. The process will focus on agriculture and explicitly consider: immediate, short and long term horizons; more/less favored areas in terms of agro−ecological and socio−economic infrastructure; direct and indirect benefits to the vulnerable.

This research will also contribute directly to capacity strengthening/building primarily at the local scale at a minimum of three study sites (Yebrage, Choke Mountain and Blue Nile Basin) through improving participating individuals, organizations and systems ability to utilize knowledge more effectively, efficiently and sustain−ability in addressing local, regional and national priorities that will contribute to adapting to climate change. The process will systematically identify and share lessons with key decision makers for further capacity strengthening/building, which is a paramount importance, to enhance innovations and adapt to climate change in ways that benefit the most vulnerable.

1.2 Rationale/Justification of the study:

Over 90% of East Gojjam population lives in rural areas and draws its livelihoods mainly from smallholder production systems centered on agriculture. However, over half of the zone's population is known to live on less than one dollar per day, with

evidence that the number of those living in poverty grew by 50% over the past 30 years. It is therefore of a major concern that East Gojjam is the only region in the world where the number of poor people is growing, a problem aggravated by an accompanying decline in per capita food production (FAOSTAT, 2006) but the minimally acceptable standard of living differs from country to country. The New Partnership for Africa's Development (NEPAD) has identified agriculture as the engine for economic growth (NEPAD, 2001; Baiono et al., 2006), and this is reflected in its Comprehensive Africa Agriculture Development Programme (CAADP).
The major challenge is therefore to reverse the current downward spiral in agricultural productivity by transforming the predominantly smallholder farming into sustainable, competitive and resilient production systems. Currently, poor and declining soil fertility (Baiono et al., 2006) remains a major biophysical cause for the recorded decline in agricultural productivity (Scherr & Yadav, 2001; Mapfumo & Giller, 2001).

Fertilizers are one of the essential inputs for maintaining or increasing the soil fertility level in intensive agricultural systems. The purpose of applying fertilizers is primarily to supply the crop with essential plant nutrients to ensure normal plant growth. The major plant nutrients (N, P and K) have to be applied regularly to compensate for the amounts exported from the soil during harvest.

In agriculture the production of plant species through sexual as well as vegetative means is of prime importance to continuity of plant generation. Most of the plant species perpetuate through sexual method of propagation, which is easy and plays a vital role in the development of new species that are best suited to the changing environment.

Every year more and more agricultural land is being converted into Eucalyptus forest (Monteith, 2007) plantations (Teferi et al., 2010; Baiono et al., 2006) which ensure income security for local smallholder residents and also prevent (Directorate−Agricultural & Information−Service, 2008) soil erosion. This is compounded by lack of access to agricultural input and output markets (LEISA, 2004), poor agricultural trade policies and introduction of different forms of economic structural adjustment programmes that have arguably disrupted or removed traditional safety nets and institutions that supported them. In many cases, these have reversed urban−rural (Gete et al., 2007) remittances and subsidies, at the expense of the natural resource base.

Millions of East Gojjam's community, both rural and urban, are therefore trapped in a vicious cycle in which poverty (Gete et al., 2007; Gill et al., 2011) is accelerating a degeneration of the natural resource base and its associated farming systems, which in turn reinforce poverty. Emerging evidences suggested that the potential impacts of climate change (Simane et al., 2012) across East Gojjam are highly variable (Fischer et al., 2006), and not only threatened to increase poverty, but also impeded efforts towards the developmentof sustainable livelihoods.

Recent experiences, in "Yebrage"and its surroundings, of severe droughts followed by flooding in the successive seasons have shown how vulnerable local communities can be to variations in climate. To avoid loss of the gains so far made in providing sustainable livelihoods, there is a need to quantify such vulnerability for the livelihood options available to different social groups within communities in major climatic zones of East Gojjam, and to establish mitigation measures.

Currently there is little empirical evidence on the state of resilience among rural communities, and their associated urban populations, to the pressures imposed by intricate factors of a degrading land resource base, weakening (Gete et al., 2007) traditional and indigenous institutions, lack of capacity to adapt new technical options, poor market access and constraining policy environments (Nielsen, 2000), in the wake of imminent impacts of climate change (Simane et al., 2012). Based on existing knowledge and previous observation on farm typologies the researcher focuses on defining the vulnerable groups and developing options to enhance their adaptive capacity.

In the light of the above, the selection of the topic was prompted not only by the desire to study an aspect of the entire observations, but also by the desire how to contribute empirical (practical) interest in view of possible applications to the technology of extraction and exploration of a variety of industrial, metallic, cement minerals, and geothermal energy, natural gas and coal deposits at feasibility levels and await development in order to eradicate the rooted poverty and hunger from East Gojjam/Ethiopia. The specific objective of the study is therefore to determine the contribution made by the researcher to forming the personality amongst the members of this community.

It seems that this short term recommendations have not been implemented, probably due to some policy implications such as committing (Gete et al., 2007) budgetary allocation and political commitment to the Zone. It thus seems clear that communities need to be empowered by being informed and being enabled to support themselves in the face of an erosion. Soil erosion occurs (Kim et al., 1996; Warren et al., 2001; Bewket, 2003; Baiono et al., 2006) when soil is removed through the action of wind and water (Konz et al., 2010) at a greater rate than it is formed. The importance of empirical study such as this cannot be refuted in such circumstances.

The farmers are losing their valuable lands (de Oliveira Camargo et al., 2010) to the agents of erosion which contributes to a significant amount of soil loss each year. Soil erosion (Bewket, 2003; Baiono et al., 2006; Gete et al., 2007; Nyssen et al., 2007) is a major (Nielsen, 2000) environmental/agricultural threat to the sustainable and productive capacity of agriculture.

This study will provide this much needed guidance in the form of identifying viable and equitable share of farmland and more sustainable agricultural service to the rural poor. The research findings will be used in augmenting the development of an overall strategy for (Gill et al., 2011) sustainable use of natural resources, biodiversity conservation and land use, depending on the proposed zonation pattern.

Based on a logical and sequential research methodology to assess availability, accessibility, affordablity, reliability and continuity of usage of fertile farmland, this study assess the current levels of nutrients in agricultural farmland for rural poor and identifies challenges and policy measurement to improve well−being access.

1.3 Purpose and Objective of the Study:

1.3.1 Research Aim:

The aim of this study is to assess the use of the NAA in order to undertake "Elemental Analysis of Selected Geological Samples" from specific areas of "East Gojjam" Administrative Zone. It is almost always better to focus on a limited area so that one can do a thorough research, rather than having a broad focus with inevitably superficial results.

1.3.2 Research Objective:

The main derive behind a piece of research is the desire to solve a practical problem. The objective of this research while dealing with the research questions is generally to reveal the elemental analysis using Neutron Activation Technique. Under this general objective specifically it attempts to:

1. Construct typical NAA on samples collected from selected areas of East $Gojjam$
2. Compare the results obtained with local and international data of similar studies
3. Establish the values of the existing technologies to determine elemental concentrations and sensitivities
4. Identify the advantages and disadvantages of NAA technique

5. Arrive at recommendation for good procedures based on the results of the data analysis that address the problem

6. Identify the contribution of the researcher towards the development of the region/country

These objectives have been identified as they are *SMART* objectives, that is: Scientific, Measurable, Achievable, Realistic and Time−founded (Mayhor & Blackmon, 2005). These research objectives were further refined by focusing the attentions on the following research questions.

1.4 Research Question of the Study:

This thesis would try to answer the following central research question and stemming from it sub−questions that would facilitate the intended research. These questions were necessary in shaping the direction of the research.

1.4.1 Central Research Question:

The central research question is a broad question that asks for an exploration of the central phenomenon or concept of the study. What is the effectiveness of the *NAA* to identify elemental content of selected geological samples from specific areas of "East Gojjam" regardless of oxidation state, chemical form or physical location?

1.4.2 Sub-Questions:

1. What type of *NAA* is currently available to do the research on selected geological samples collected from specific areas of East *Gojjam*?

2. What approach can be developed to rapidly construct NAA applications facilitating the gathering of vital and accurate information for the analysis of samples?

3. How the obtained results are compared with local and international data from similar studies?

4. What are the advantage and disadvantage of NAA approach?

5. How the concentrations and sensitivities of the elements under investigation are differentiated ?

6. How recommendations are formulated based on the results of the data analysis that address the problem and enhance investigations of new skills?

7. What is the contribution of the researcher towards the development of the country/region?

All these questions will be asked and the results will give larger and clearer picture on the elemental analysis of some geological samples using NAA. It is believed that in answering these questions by applying certain methodological techniques: In the first place, a certain amount of desk−based research is needed. This will involve findings and reviewing relevant academic materials, by searching library data−bases and the internet for books, journals and articles related to the topic.

The second research technique will be laboratory based with Neutron Activation Techniques. This will involve researches on activation design and on appropriate detection technique to contact. Research plan methodology will be explained in more details in the “Research Methods” section (sea the third chapter).

1.5 Significance of the Study:

In developing countries, harsh climatic conditions, population pressure, land constraints, and the decline of traditional soil management practices have often reduced soil fertility. Because agriculture is a soil−based industry that extracts nutrients from the soil, effective and efficient approaches to slowing that removal and returning nutrients to the soil will be required in order to maintain and increase crop productivity and sustain agriculture for the long term. The overall strategy for increasing crop yields and sustaining them at a high level must include an integrated approach to the management of soil nutrients, along with other complementary measures.

An integrated approach (Gruhn et al., 2000) recognizes that soils are the storehouse of most of the plant nutrients essential for plant growth and that the way in which nutrients are managed will have a major impact on plant growth, soil fertility, and agricultural “sustainability”.

Farmers, researchers, institutions, policy makers and politicians all have an important role to play in sustaining agricultural productivity. Effective and efficient management of the soil storehouse by the farmers is thus essential for maintaining soil fertility and sustaining high yields. To achieve healthy growth and optimal yield levels, nutrients must be available not only in the correct quality and proportion, but in a usable form and at the right time.

Flooding within East *Gojjam* may be equally devastating, but often more localized than droughts. It may cause soil erosion/soil movement, damage (Hartemink, 2003) to crops, rotting the roots and killing the plant, damage to roads and infrastructure and damage to animal and human lives in extreme situation. Limited or decrepit infrastructure increases the possibility for loss of life during flood conditions.

Erosion is the process of detachment and transport of soil particles by erosive agents (Baiono et al., 2006). Soil erosion over farmlands of East Gojjam is a (Ustum, 2008) quite−frequent and well−distributed problem and occurs when soil is removed through the action of wind and water (Baiono et al., 2006) at a greater rate than it is formed.

The soil covering the surface of the earth has taken millions of years to form. This means that soil is a non−renewable natural resource and once destroyed it is gone forever. If this is disregarded, a time will come when there would not be enough soil left to sustain life on earth, because the soil is a necessary growth medium for plants, a home for certain insects and animals, as well as a medium from which we get minerals. It is important therefore to treat soil, especially topsoil, as a living entity. Plants (Monteith, 2007) break the impacts of a raindrop before it hits the soil, reducing the soils ability to erode.

Causes of soil erosion (Konz et al., 2010) occur when farming practices are not compatible with the fact that soil can be washed or blown away. These practices are overstocking and overgrazing, inappropriate farming techniques such as deep ploughing land 2 or 3 times a year to produce annual crops, lack of crop rotation, planting crops down the contour instead of along it, lack of proper land use system. Much of the eroded soil is deposited in low areas of the field, or it moves off the farm and eventually enters drainage ditches, streams or river called *Chemoga* (Getachew, 2003; Teferi et al., 2010), which is a major tributary of Blue Nile River. Soils that enter this watercourse reduce water quality (Stuart, 1971), efficiency of drainage system and storage capacity of the river.

1.6 Contribution to new knowledge:

One of the contribution of the study to a new knowledge is related to the use of (Santoso et al., 2010) nuclear techniques in fields such as agriculture, health, industry, and research support environmental objectives. The followings are some examples of these areas: Nuclear techniques are used to trace fertilizers, to determine the best form, timing, placement, to avoid waste and reduce its movent into the environment. Others are used to detect, measure, and track fertilizers supplied nutrients in soil and plants (soil fertility and crop production), and they (Santoso et al., 2010) play a key role in investigating the environmental consequences in clearing of the tropical rainforests.

Nuclear techniques using "Environmental Isotopes" are among those that meteorologists, hydrologists, and hydrogeologists use in the study of water.

NAA plays also a leading role (Chung et al., 2004) among nuclear analytical techniques in the field of environmental research related to geological samples and human health. Because of their sensitivity, stable isotopes can measure small changes over short period of time, environmental and ecological changes caused by relatively small cleared areas and basins.

The other contribution to new knowledge made through the study is the development of an assessment tool which has already been tested and found to be useful in preventing soil erosion/movement.

The appropriate use of external input paradigm, which was deriving the research and development agenda, namely fertilizers, lime, or irrigation water, was believed to be able to alleviate any constraint to crop production. Following this paradigm together with the use of improved cereal germplasm, the application of "Green Revolution" strategy, which boosted agricultural production in Ethiopia, results in minor achievements.

Currently, soil fertility research and strategy focus on the new paradigm of Integrated Soil Fertility management, which is a holistic approach in soil fertility research that embraces the full range of driving factors and consequences of soil degradation, biological, chemical, physical, social, economic, health, nutrition, and political.

1.7 Delimitation of the Study:

The study area is delimited on "East Gojjam Administrative Zone". The land holding, usage and management system of East Gojjam is traditional, backward, rain−fed, unplanned and unmechanized which is characterized as belonging to all the descendants of the ancestors. They use the land communally and because of this there is a strong attachment to the land. The absolute ownership of the land rests in the hands of the settlers. The principleof father to child inheritance resulted in uncontrollable measure of *parcellation* and fragmentation.

The community depend on annual rain−fed crop production of which "teff" (Eragrostis Teff), barley, wheat, cereals, beans, peas, **Gibto (Lupinus Albus)** and the likes. Most of the farmlands are mountainous and some are flat. The geographical locations, its topography and the climatic conditions are somewhat comfortable because its mean annual temperature is $(15 - 20)$c°.

1.8 The Structure of this Work and Content Analysis:

The chapters of this thesis will be organized as follows. To better understand the processes at work in retaining "Elemental Analysis of Some Geological Samples using Neutron Activation Technique":
Chapter one discusses the introduction to the research that provides the background and problem definition, the rationale/justification, research aim and objective, research questions, the study area and significance of the problem, and also the role of nutrients in creating and enabling environment for plants to grow.
Chapter 2 examines the nuclear instrumentations. The researcher would describe the available neutron source and detection system, explain how neutron activation analysis compares to others and show some of the typical applications. Finally, the researcher would explain the experimental framework needed to NAA. This included the basic steps in the irradiation and acquisition process, and the software that is developed for this purpose.

Chapter 3 examines the methodology, the data gathering techniques used, and the data analysis information. It would describe the software that is used for the analysis of the data generated during the experiment. It represents a major part of the effort that went into this research. An overview would be given of the features and functionality specially for the conducted experiment.

Chapter (4, 5, 6, 7) discusses many of the experiments, possible results and findings in elemental analysis at the sound level. It would give an extensive description of the scientific experiments performed for this doctoral thesis. The aim of these experiments was to further develop the possibilities of Neutron Activation Techniques.

Finally Chapter 8 draws the results and conclusions of the preceding chapters and offers some recommendations for future studies in improving the management of plant nutrients and soil fertility in the years ahead. A list of references followed at the end of these chapter.

1.9 Summary:

The loss of protective vegetation through overgrazing, ploughing and fire makes soil venerable to being swept away by wind and water. Plants provide protective cover on the land and prevents soil erosion for the following reasons. Plants slow down water as it flows over the land and this allows much of the rain to soak into the ground and also break the impacts of a raindrop before it hits the soil, reducing the soil's ability to erode. Plant roots hold the soil in position and prevents it from being blown or washed away.

Crop rotation is a device/or an effective means for maintaining high soil productivity over several years of continuous cropping and controlling diseases and pests. The inclusion of legumes and fallow in the cycle and the use of crops that feeds at different levels are devices to sustain soil productivity.

Mixed cropping stabilizes the environment and protects the soil from hazard like high rainfall intensity which results in the erosion of land, loss of nutrient and high temperature which leads to high rate of mineralization.

Soil organic matter (SOM), which is the "glue" that binds the soil particles together and plays an important part in preventing soil erosion, is that fraction of the soil composed of plant and animal remains in various stages of decomposition and synthesis including cells and tissues of soil organisms, and substances from plant roots and soil microbes.

Nuclear techniques are used to trace fertilizers to determine the best form, timing, and placement to avoid waste and to reduce its measurements. Others are used to detect, measure, and track fertilizer−supplied nutrients in soil and plants, determine the ability of soil moisture, study and promote the natural processes of nitrogen fixation.

Based on sound fundamental theory, the research leads to a practical consideration of soil as a living system in nature and illustrates the influence of human activities upon soil structure, texture (Moeys, 2011; Berry et al., 2007; Bormann, 2007; Giddings,

2004) and function. Researchers, as well as other readers, will better understand the importance of soils and the pivotal position theory occupy with respect to careful and knowledgeable conservation.

Chapter 2

Theoretical Background:

This Chapter aims to provide a theoretical background to the empirical research and provides a critical evaluations and interpretations and relevant instrumentations pertaining to the scope of this study.

2.1 Introduction:

Choosing the scientific measurements is crucial for getting the correct conclusions. Some measurements might not reflect the real world, because they do not measure the phenomenon as they are (Experiment−Resources, 2008b).

2.1.1 Neuclear Instrumentation:

The radioisotope production can be performed through different nuclear reactions, which could take place in isotopic neutron source. The instrumental neutron activation analysis (INAA) (Araripe et al., 2006; Kahtani et al., 1986; Sarathi et al., 2008; Amin et al., 2010) was carried out using isotopic neutron source produced by

($Am^{241} - Be^{9}$) (Filby, 1995; Daraban et al., 2005) facility with the source located in the center of a paraffin howitzer. In Neutron Activation Analysis, a sample of material (Gaudreau & Hardin, 1974) is bombarded with neutron flux and is made radioactive.

All irradiations (Daia et al., 2001) of samples together with their standards were carried out in the facility of thermal neutron flux for a certain irradiation time. As the radioactive atoms disintegrate, high energy gamma and other rays are emitted, these rays are counted and analyzed. Activation analysis (Sansoni, 1987; Avino et al., 2007) is a method of elemental analysis using techniques of nuclear transformation.

Neutron Activation Analysis (NAA) was discovered in 1936 when Hevesy and Levi (Win, 2004) found that samples containing certain rare earth elements became highly radioactive after exposure to a source of neutron.

NAA (Brown & Milton, 2005; Sarathi et al., 2008; Chung et al., 2004), which is a very powerful analytical technique, determines elemental content and concentration (Avino et al., 2007; Srinivas, 1995; Sarathi et al., 2008) of many elements present in samples (Hassan, 2008; Cristache et al., 2008; Essiett et al., 2011) of unknown compositions. It performs both qualitative and quantitative multi−element analysis of (Win, 2004; Abd et al., 2009; Kogo et al., 2009; Essiett et al., 2011; Berlizove, 2006) major, minor and trace elements regardless of their oxidation state, chemical composition or physical location and converts some atoms of a sample's elements into artificial radioactive isotopes (Wadley & Kempson, 2011) by irradiation with neutron.

An isotope of atomic mass A and atomic number Z (Kasban et al., 2011) when placed in a neutron flux will, in general, absorb a neutron to become the isotope (A+1, Z). Some resulting isotopes, being unstable, will decay, emitting one or more gamma−rays with energies and half−life characteristic of the particular isotope. During irradiation the naturally occurring stable isotopes of most elements are transformed into radioactive isotope by neutron capture. The number of radionuclide produced in activation of a natural isotope is given (Win, 2004) by the expression:

$$N_{A+1} = \frac{\phi \sigma_{aA} N_{aA}}{\lambda_{A+1}} \left[1 - e^{-\lambda_{(A+1)}T}\right] e^{-\lambda_{(A+1)}t} \quad (2.1.1)$$

And the activity

$$A = \frac{dN_{A+1}}{dt} \quad (2.1.2)$$

where, ϕ−is the neutron flux, (σ_{aA})−is the neutron absorption cross−section of the original isotope, (N_{aA})−is the number of nuclei of the element under consideration of the isotope, (A, λ_{A+1})−is the decay constant of the created isotope, T−is the irradiation time and, t−is the time between stop of irradiation and start of the counting of the activity.

The activity of a radioactive source is the number of atoms decaying per second. The half−life of a radionuclide is the time it takes to lose 50% of its activity by radioactive decay.

The (NAA) is accepted as an important technique (Dias & Prudêncio, 2007; Santoso et al., 2010) for elemental analysis of geological samples. This will serve in the local industry and economy in each regional area.

2.1.2 The neutron source:

The (α, n) source in which an alpha (α) emitter, such as Am^{241} is mixed with target Be^9, produces neutron source (Fedoroff et al., 1995). The capture of a neutron (Sadiq et al., 2010) results in the emission of a $\gamma-$ray. The (Daraban et al., 2005) isotopic neutron source of ($Am^{241} - Be^9$) (Filby, 1995; Karadag et al., 2003) type was used for some radionuclide production. This isotopic neutron source has an activity of 2 Ci mounted on 3 cm by 3 cm area. This source is introduced in a closed$-$end tube which is placed inside (Fischfeld et al., 2004) a paraffin block. The usefulness of this

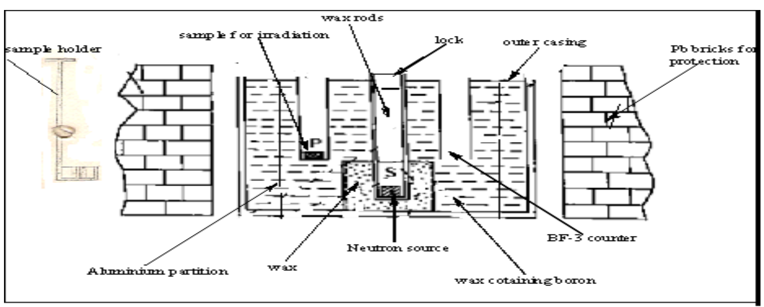

Figure 2.1: The irradiation block containing the Am$-$Be neutron source

source for such studies is very limited due to the limitation of both of the flux and the activity of the beam. This neutron source found in nuclear physics laboratory is very weak compared to that of nuclear reactor source. The (n, γ) process has been observed in nearly all of the elements.

The formula used for the calculation of the radionuclide activity is (Daraban et al., 2005):

$$\Lambda = \frac{A}{t.\epsilon.p} \tag{2.1.3}$$

Where $\Lambda-$ represents the activity in μCi, A−the area of the peak, $\epsilon-$the efficiency, p−the probability of transition at this particular energy as a result from the disintegration scheme, t− represents the acquisition time.

Neutron Flux:

The neutron flux "ϕ", which is a very significant quantity in all studies concerning Neutrons (Shiraki & Liu, 2005; Miller et al., 2004; Maayouf et al., 2008; Jie et al., 2003), is the amount of neutrons available for irradiation and can be expressed as:

$$\phi_{th} = \frac{(dN/dt)Ae^{\lambda t_d}}{\epsilon_G I_\gamma K \sigma m N_A f(1-e^{-\lambda T})(1-e^{-\lambda t_m})} \tag{2.1.4}$$

Where, t_m-real time measurement, T−irradiation time, t_d −transporting time, ε_G-geometry dependent efficiency of the detector, $\sigma-$thermal neutron capture cross−section, $\lambda-$decay constant of product nuclei, A−the atomic weight of the element, m−mass of the bombarding element, $\frac{dN}{dt}-$the activity of the emitted gamma, N_A-Avogadro number, f−the isotopic abundance of the target nuclei, $I_\gamma-$the intensity of the gamma ray line and K−the self absorption of gamma rays in the sample.

The same expression of $eq.$(2.1.4) can be used for the determination of mass of the unknown element present in the experimental sample.

Activation is possible to induce radioactivity into materials by letting them interact with radiations. As soon as the irradiation starts, the radioisotope starts decaying. This means that both the process, irradiation and decay, are happening at the same time.

The process of irradiating a material with neutron makes the material itself to be transformed into a radioactive nuclide.

Depending on the neutron flux energy spectrum and reaction cross−sections, the target nucleus undergoes a nuclear reaction and the resulting nucleus will immediately de−excite under emission of characteristic prompt gamma−rays into a more stable configuration. This configuration is in general a radioactive nucleus with a certain half−life ($t_{\frac{1}{2}}$) which will further decay under emission of characteristic delayed gamma−rays into a stable product nucleus. An illustration in the case of a neutron capture reaction is depicted in fig.(2.2). The (n, γ) reaction is the fundamental reaction for neutron activation analysis (Descantes et al., 2008).

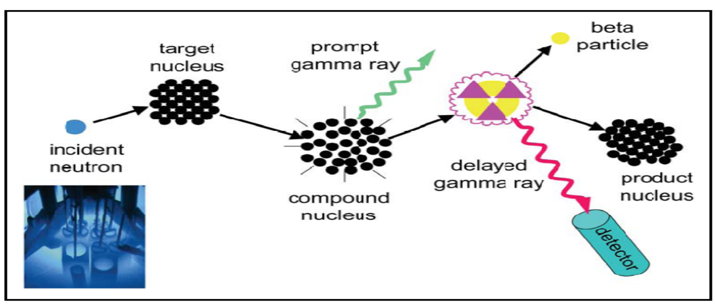

Figure 2.2: Schematic diagram of neutron capture

2.1.3 Detection Techniques: Apparatus and equipment:

Radiation is detected using special systems which measure the amount or number of ionization or excitation events that occur within the detector's sensitive volume. In a semiconductor detector, the information carriers of the radiation are the electron-hole pairs.

Quantitative measurement of radioisotopes is made at NAA Laboratory by using gamma spectroscopy instrumentation system:

HPGe detector/Spectroscopy/Amplifier/Multichannel Analyzer/Genie−2000 software system. A gamma−ray spectroscopy (El-Taher, 2006) system (Robu & Giovani, 2009) consists of a detector (and high voltage power supply for the detector), pre−amplifier, spectroscopy amplifier, <analog-to-digital>converter, multi−channel analyzer, and an output device.

A high−resolution gamma−ray spectrometer is used to detect those delayed gamma−ray(s) in the presence of artificially induced radioactivity in the sample for both qualitative and quantitative analysis (Win, 2004).

In order to minimize thermal noises, the detector is kept at cryogenic temperatures (liquid nitrogen, temperature=77K). The initial signal is very small and the pre−amplifier, attached directly to the detector, amplifies this signal. The signal is shaped by the spectroscopy amplifier and then converted from an analog to a digital signal by the <analog-to-digital>converter. The results are stored in digital form (multi−channel analyzer). The small box on top of the cabinet combines all the functions of the HV power supply, spectroscopy, amplifier and<analog-to-digital>converter.

The resultant spectrum is shown on the computer screen. A computer is used to visually show the resulting spectrum and to do calculations on the spectrum. The use of computers connected to more (Gaudreau & Hardin, 1974) sensitive counting equipment has enabled activation analysis to be employed successfully in diverse areas of research and applications.

Various algorithms are used to determine the shape and energy of each gamma−ray peaks present in a spectrum and to determine the area encompassed by the peaks (i.e., the gamma−ray intensity). Subsequent decays, interferences (if required), fluence, fission product corrections, and comparison with a standard lead to a quantitative analysis.

The apparatuses are normally left fully connected on the laboratory bench, so that no need of rewiring them without good reasons.

2.2 Experimental setup:

The basic instrumentation for performing neutron activation analysis consists of a nuclear source (Jack et al., 1999) for irradiation the samples and standards (Daia et al., 2001), nuclear detector for detecting the gamma−emissions, and various types of multi−channel analyzer system that range from simple data acquisition systems to complex computerized data acquisition and processing systems. Each irradiated sample was measured with a gamma−ray spectrometer (Srinivas, 1995) consisting of ($HPGe$) set up and a multichannel analyzer.

Outside the vacuum chamber, to analysis the signal from the detector, a conventional electronic chain made up of a pre−amplifier (PA), an amplifier (A), an <analog-to-digital>converter (ADC), a multichannel analyser (MCA) and a system for data acquisition (Genie−2000) was used.

The detector is shielded in a number of layers starting with stainless steel (5 mm thick) and lead (30 mm thick) to reduce the background radioactivity on all sides. Lead is a well (Ljung et al., 2011) documented neurotoxic metal. Because of its high density and large atomic number, lead is the most widely used material for the construction of detector shields (Knoll, 2000). The detector used (Karadag et al., 2007) in a measurement was high−purity germanium, manufactured by Canberra Inc.

Shielding is a protective barrier, usually a dense material, which reduces the passage of radiation from radioactive materials to the surroundings.

The function of the detector is to produce a signal for every particle entering into it. Every detector works by using some interaction of particle with matter. The Data acquisition system (Kasban et al., 2011) is used to collect the data from the detector on a computer. With this detector, more than one radioisotope can be detected at the same time.

The details of procedural steps carried out during the course of experiments and various parameters studied are given below. The schematic diagram shows the instrumentation part used in NAA Laboratory.

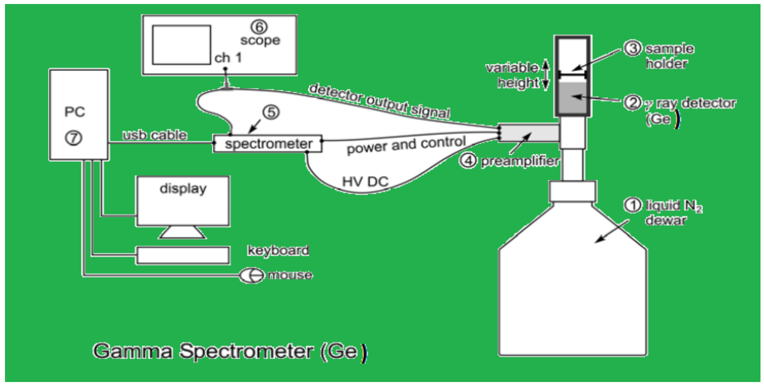

Figure 2.3: The experimental arrangement

1. **Detector**: The high purity germanium diode is mounted in a cryostat consisting of a vacuum chamber thermally coupled with liquid Nitrogen (77K) heat sink. Ge (El-Taher, 2006) crystals have inherently good energy resolution characteristics, having relatively high atomic number which helps to stop high energy gamma radiations and it can be refined to an extremely high degree of purity. Here the gamma$-$ entering the detector cause ionization of the Ge crystal, thus generating a small signal, the magnitude of which is proportional to the gamma photon energy.

2. **Pre-Amplifier**: It maintains low degree of noise and high degree of stability.

3. **Spectroscopy amplifier**: Because of long decay time constant pulses from pre$-$amplifier are vulnerable to pile up distortion effects i.e. the rise of one pulse is distorted by the tail of the preceding one. This is minimized by this amplifier and adequate signal to noise ratio is maintained. Here 3000 volts electric field is applied to collect the liberated charge from the detector.

4. **Multi-Channel Analyzer (MCA)**: It consists of three parts, an analogue to digital converter (ADC), Data scaler & Memory accumulator. *ADC* converts the analogue Gaussian pulse from the main amplifier to digitized signal. *MCA* sorts out all these pulses and displays the results in the form of a graph on the (PC) Monitor. Using a suitable computer programme, the area of a particular energy peak can be found out and the concentration of the element can be determined.

Methodology for INAA: It is a multi−step process and involves the following steps:

1. **Planning**: A successful and accurate analysis depends upon the planning and careful execution of subsequent steps in the analysis.

2. **Preparation of sample for irradiation**: After weighing accurately of homogenous and representative sample of soil/rock, it is carefully sealed in polythene sheet for irradiation at Isotropic Neutron Sources.

3. **Irradiation time**: This depends upon the half lives of elements determined and the amount of the element present.

4. **Cooling**: After irradiation samples must be cooled until it is safe to handle and reduce high activity of short lived elements from geological samples. This also helps to keep dead time of counting system below permissible level.

5. **Counting**: The number of radiations detected per unit time is called counting rate. Initially the instrument is calibrated with known reference standards. Two counting are carried out first for short lived elements, second counting for short & long lived elements.

6. **Results computation**: Basic spectrum analysis is carried out by Personal Computer (PC) based on MCA with spectra display on monitor and by identifying the specific isotope from gamma energy & concentration determined by comparing the net areas of the photo−peaks with those of standards. Peak area is determined by Genie−2000 software with the help of computer. Results are reported in (ppm) & (ppb) as per requirement.

The concept of (INAA) (Amin et al., 2010) is to produce radioactive isotopes by exposing the samples to a high flux of (Daraban et al., 2005) neutrons in isotopic neutron source. The pulses between the pre−amplifier and amplifier generally have a

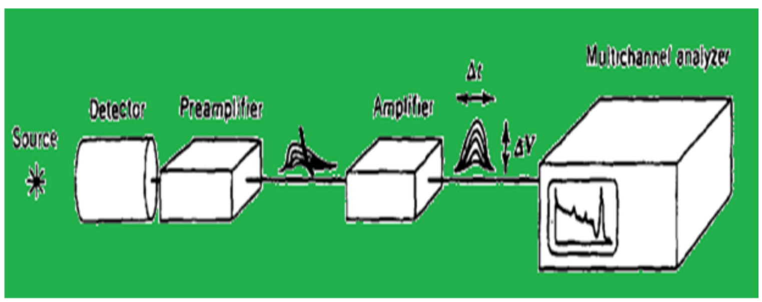

Figure 2.4: Schematic Diagram of electronic equipment that might be used in a measurement of the energies of radiations emitted by a source

short (nanosecond) rise time and a long (millisecond) decay time, with an amplitude of millivolts. The amplifier pulses are more symmetric, with a width of Δt of μs of a few volts. The multichannel analyzer display shows Δv of the horizontal axis.

Observing pulses from the detector: When turning <ON>the spectrometer, notice that the High Voltage light is green and this indicates that the detector is ready but that no high voltage is being delivered yet. The Analyzer is continuously making digital measurements of amplitude at a fast rate, differentiating the measurements over a 1 μs interval and recording any cases where the differences are between the lower and upper limits. The recorded events are displayed as histograms on the screen.

Observing a Cs^{137} spectrum: In order to start gathering data with the Genie−2000 Spectrum Analyzing Software, it is important to understand how the software interacts with the output from the detector.

Note that the sum peak that is seen with Cs^{137} has different shape with that of Co^{60} (Nakamura et al., 2001, 2003).

2.2.1 Calibrating of the Spectrometer:

For calibrations, the standard source is placed above the detector in a well defined geometry, and the measurement is started. $HPGe$ gamma spectroscopy detection systems are generally operated on an energy range of 0 to 2000 kev utilizing 4096 to 8192 channels of data acquisition storage. The three calibration items of daily interest: energy calibration, detector peak resolution and efficiency stability (Jack et al., 1999). The dependence of the efficiency on the radiation energy was determined at 0.0 mm sample−detector−distance. The detector efficiency decreases continuously with energy. It can be noticed that the detector efficiency decreases with the volume of the sample in the energy−range of interest.

To minimize the effect of the scattered radiation (Win, 2004) from the shield, the detector is located in the center of the chamber. The spectra were either evaluated with a computer software program, or manually with the use of a spreadsheet to calculate the concentrations (Arakawa et al., 2003) of the elements.

The neutron activation analysis system/or spectrometer was calibrated for efficiency and energy using 10 standard gamma−rays of Eu^{152} source, one gamma−ray of Cs^{137} source and one Co^{60} source. The standard sources peaked in the same geometry as that used for measuring gamma−rays from samples. The spectrometer should be calibrated once per week or before use if the meter has stored for more than a week.

To perform the calibration, standard radioactive point sources which emit gamma−rays of energies are used as shown in the following table. In order to calibrate the spec-

Table 2.1: The three standard sources

isotope	half-life(year)	Energy(kev)
Cs^{137}	30.17	661.661
Co^{60}	5.271	1173.24
		1332.51
Eu^{152}	13.537	121.3
		244.4
		343.6
		443.7
		777.7
		865.9
		962.1
		1084.3
		1110.7
		1405.7

trometer, it is needed to identify which peak belongs to which gamma−ray emitter. To find the correct channel value corresponding to each peak, it is needed to define a Region of Interest (ROI).

2.2.2 Energy Resolution:

The most important characteristics of $HPGe$ detector is its excellent energy resolution. The great superiority of the $HPGe$ detector allows the separation of many closely spaced gamma−ray lines, which are very beneficial for measuring multi−gamma emitting radioactive sources. The energy resolution of the $HPGe$ photon spectroscopy system is governed by the variation in the number of charge carriers, variations in the charge carrier collection, and the contribution of the electronic noises. The higher energy resolution of a semiconductor detector compared to a scintillator is due to the larger number of carriers for a given incident radiation event.

Detection Limit: Detection limits (IAEA, 2001; James & Cynthia, 2002; Nassef et al., 2008; Romoli et al., 2005) vary from element to element because the sensitivity of the method for a given element is partly a function of the reaction cross−section and the half−life of the induced radionuclide.

2.2.3 Detector Energy Calibration and Analytical Data:

In order to convert the electric signal of the detector (channel) to energy (kev), an energy calibration must be performed. The software used to analyze the germanium crystal detector signals in this experiment was a Canberra product called Genie−2000 and it was used to measure the centroids and areas of each peaks. In gamma−ray spectroscopy with Germanium detector, the pulse height scale must be calibrated in terms of absolute gamma−ray energy if various peaks in the spectrum were to be properly identified.

Accurate calibration (Jayaseelan et al., 2010) should involve a standard source with gamma−ray energies that are not widely different from those to be measured in the unknown spectrum.

The precision to which the centroid of a peak in a pulse height spectrum can be localized is dependent on the spectrometer resolution system and its stability over a period of the measurement. Therefore, an important goal is to define closely the energy of the standards so that their energy uncertainty did not contribute unnecessarily to the overall imprecision of a gamma−ray measurement. The maximum intensity peaks of

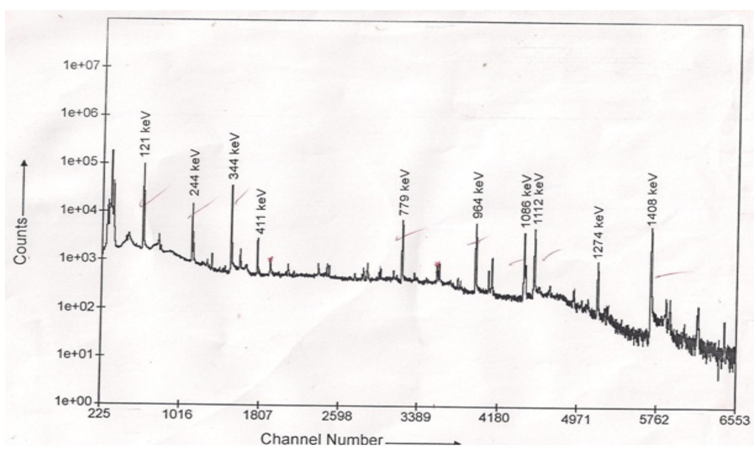

Figure 2.5: A typical gamma-ray spectrometer of Eu^{152} source

121.8 Mev is conjugated channel 244, internal calibration energy is 122 kev.
Once energy calibration points have been established over the entire energy range of interest, a calibration curve relating energy to channel number was normally derived.

Gamma−rays incident from a specific direction would tend to interact preferentially

Table 2.2: Energy Calibration

Energy calibration		
standard sources	channel	energy(kev)
Cs-137	1376	661.6
Eu-152	244	121.8
	688	344.3
	2812	1408
	1924	964
	2216	1112
	1556	778.9
	2172	1086
	488	244
	1732	867.4
	888	444

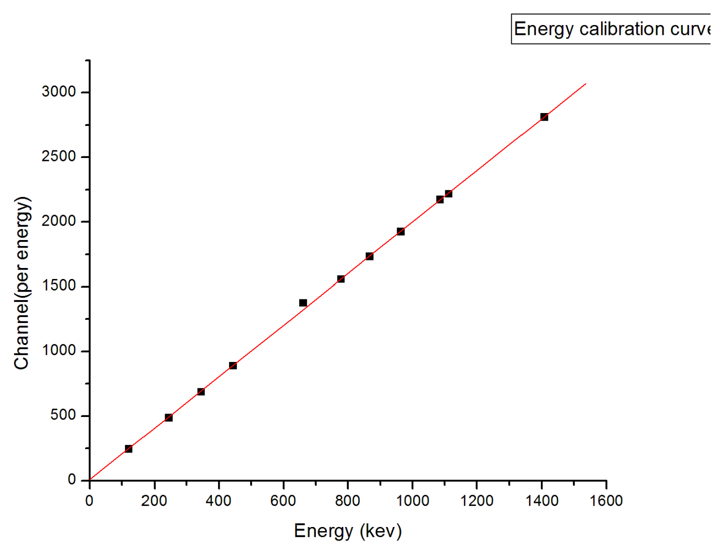

Figure 2.6: Energy calibration curve

in certain regions of the detector volume. If the charge collection efficiency varied appreciably between regions, then the average peak position could also shift with incident gamma−ray directions.

2.2.4 Detector Efficiency Calibration and Analytical Data:

Any measurement of absolute emission rates of gamma−rays requires knowledge of the detector efficiency. The emission rate for a point source can then be calculated by measuring the full−length energy peak area over a fixed time and by determining the detector solid angle from its dimensions and the source−detector spacing.

In germanium gamma−ray spectroscopy, an efficiency based on the area under the single or double escape peak is sometimes used in place of that based on the full−energy peak.

The <source-detector-distance>still must be accurately (Jayaseelan et al., 2010) measured and reproduced to avoid errors in the relative solid angle. The calibration is normally carried out for an assortment of gamma−ray energies covering the range of interest to allow construction of an empirical efficiency versus energy curve.

Due to detector efficiency (Leinweber et al., 2000) is dependent on several factors, including the composition of the standard, the geometry of the sample, and its position in relation to the detector, it is meant that the single valued of detector efficiency of a specific energy can only be used for the same geometry.
Fig.(2.7) shows the (%) efficiency of the four possible <source-detector-distance>at various energies.

Table 2.3: Efficiency Calibration

Efficiency× 10^{-5}				
energy(kev)	at 0 cm	at 5 cm	at 7 cm	at 10 cm
121.3	117.75	26.46	17.94	10.75
244.4	87.41	19.13	13.41	8.49
343.6	78.75	15.15	10.60	6.35
443.7	60.20	13.11	8.29	6.86
777.7	35.94	7.95	5.96	3.30
865.9	29.45	5.41	4.73	2.42
962.1	34.90	6.43	4.45	2.56
1084.3	38.84	6.09	4.39	2.58
1110.7	34.04	5.68	4.57	2.17
1405.7	26.15	4.96	3.27	2.18

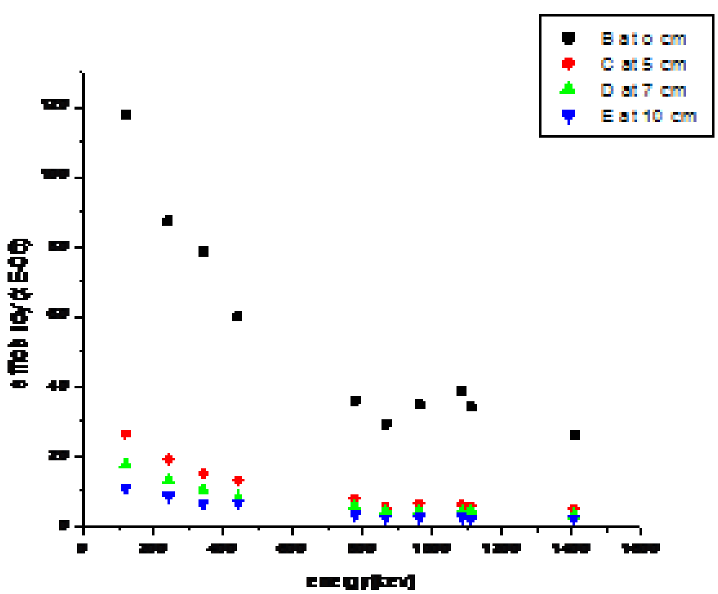

Figure 2.7: Detector efficiency curves

Calculation for efficiency at different gaps:

$$\epsilon_G = \frac{1}{t.N}\frac{C}{\theta} \tag{2.2.1}$$

where t−counting time, C−area of the peak, θ−yield

Once the efficiency of a detector has been measured at several energies using calibrating sources, it is useful to fit a curve to these points that describe the efficiency over the entire energy range of interest. Efficiency of the detector (Leinweber et al., 2000) is dependent on the energy of each gamma−ray.

2.3 Irradiation and Acquisition:

A sample is subjected to a neutron flux and radioactive nuclides are produced. As these radioactive nuclides decay, they emit gamma−rays whose energies are characteristic for each nuclide. Comparisons of the intensity of these gamma rays with those emitted by a standard permit a quantitative measure of the concentrations of the various nuclides.

Neutron Activation Analysis (Michael & Hector, 2003) is one of the most sensitive and powerful technique for performing non−destructive (Vega−Carrillo et al., 2006), rapid, quantitative, simultaneous multi−elemental analysis (Berlizove, 2006). It requires irradiation of geological samples at isotopic neutron source by (n, γ) type reaction. The radioactive nuclides produced (Win, 2004) in this activation process usually decay by emission of a beta particle (β^-) and a gamma−ray with a unique half−life.

When an isotope of a stable element is irradiated by the nuclear particles produced in an isotopic neutron source, some of the atoms of the isotope interact with the bombarding of particles and are converted into a radioactive isotope of the same element or into isotopes of different elements depending (Sansoni, 1987) on the nature of the bombarding particle.

2.3.1 Background Precautions:

Radioactive sources placed near the experimental apparatus often affect the data being taken more than being a health problem. In all experiments there are background counts which appear due to radioactivity in the building walls, in the ground, and in other sources in the room, as well as from cosmic radiations. The background radiations from sources in the room can be reduced by keeping neighboring sources far from the detector, and also by shielding the detector.

2.4 Summary:

The main radiations involved in this measurement method are neutron and gamma−rays, both uncharged, therefore characterized by high penetrations. Special care is also needed for duration of irradiation time, position in the source, geometry at the time of counting, elapsed time between end of irradiation and counting.

Instrumental Neutron Activation Analysis is a multi−element analytical procedure that is applicable to the qualitative and quantitative determination of the elements. Neutron Activation Analysis in its various forms is still active and there are good prospects in developing countries for long−term growth. This can be achieved by a more effective use of existing irradiation and counting facilities, a better end−user focus, and perhaps marginal improvements in equipment and techniques.

$INAA$ being a multi−element analytical procedure reduces the high cost resulting from reactor costs, analytical services and travel. The large number of elements and samples that can be determined simultaneously offsets analytical services if cost is determined on a per analytic bases.

$INAA$ is often non−destructive and the sample could be used for other analysis or if an important archival sample can be recovered. No discernible changes in the sample can generally be observed, however, the sample could be slightly radioactive due to the production of long half−life radionuclides.

Chapter 3

Research Methodology:

This chapter aims to explain the method used to conduct the research component of this study. This will be discussed in terms of aims set for this research, the research design, ethical considerations, subjects, material apparatus and procedures used.

3.1 Introduction:

The selection of the research method (Cohen et al., 2004) is crucial for what conclusions you can make about a phenomenon. It is also important to choose a research method which is within the limits of what the researcher can do. Time, money, feasibility, ethics, and availability to measure the phenomenon correctly are examples of issues containing the research (Experiment−Resources, 2008b). This section describes the methodology (Shuttleworth & Martyn, 2009b) which is used for this research work.

3.2 Research Methods:

It is important to have an understanding of the research methodology in order to provide a structured approach to the research process. And a research method is a way to (Kothari, 2004) systematically solve the research problem and also helpful in interpreting the findings of other research works.

3.2.1 Kind of Research:

Basically this research work on" Elemental Analysis of Some Geological Samples using Neutron Activation Technique" is basic research.

The Quantitative Research Paradigm:

A quantitative experimental method was used for this study. The method was used to contrast a picture of a phenomenon, explore events, soils, rocks or situations as they naturally occur. In this study information on the nature and extent of the use of activation and detection techniques were obtained.

3.2.2 Experimental Research:

Experimental research is a controlled investigation where certain variables are manipulated while other variables are kept constant. Experimental research is basically the method that can be applied in a research laboratory (Max, 2007). The basic structure of this type of research presupposes a cause and consequence relationships. The two situations (cause and consequence) are assessed in order to make a comparison. The experiments should be well maintained with the objective to be able to measure the effect of treatments used in the experiment.

3.2.3 Research Design:

The method provides the glue that holds the research project together. A design is used to structure the research, to show how all the major parts of the research project, the samples or groups, measures, treatments or programs, and methods of assignment work together to try and address the central research question. A research design is an exposition or plan of the way in which the researcher intends to deal with the research problem that has been formulated. The reason that a researcher design is applied to a research study is to provide the overall structure for the procedures that the researcher follows, the data that the researcher collects and the data analysis that the researcher conducts (Leedy & Ormrod, 2001). The researcher used an empirical design for this research because of limited literature available on the topic.

3.3 Experimental Work:

3.3.1 Materials and Methods:

Each sample was bagged separately in<zip-lock-bags>and placed together in a small plastic container. The samples were placed to radioactive neutron source in a shielded container for many days. The samples were withdrawn from the source and then taken to the counting room for counting.

3.3.2 Study Areas:

The study was conducted on an area of East Gojjam Administrative Zone. Specific study areas are located in North−Western side of Ethiopia: Yebrage (Debre Markos, Chemoga River basin) ($10^0 20' 02" N$) latitude and ($37^0 43' 47" E$) longitude, Blue Nile

River basin ($10^0 04' 02" N$) latitude and ($38^0 11' 24" E$) longitude and Choke Mountain ($10^0 34' 40" N$) latitude and ($37^0 50' 31" E$) longitude with an elevation of 2450, 1036 and 3950 in meter above mean sealevel ($amsl$) respectively. These research areas are almaost barren.

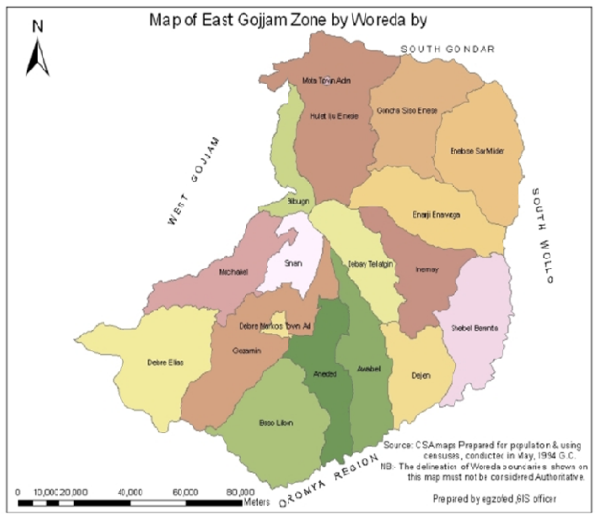

Figure 3.1: Map of East Gojjam Administrative Zone

GPS (Global Positioning System) (Maliszewska−Kordybach et al., 2008; Angelal et al., 2010) records the results of researchers on maps : the exact locations of research sites in terms of elevation above mean sea level (amsl), latitude and longitude, which are basic tools. The sample locations that were determined by the use of GPS were plotted on the geological map of the study areas. GIS (Geographic Information System) (Kijowska, 2004) collects, stores, retrieves, analyzes and displays geographic information or spatial data on earth's surface. The GPS provides accurate positioning of the sample points, so that accurate geo−referenced maps of nutrient levels can be made with geographic information system (GIS), and related to other data sets such as yield maps, soil survey, and remote sensing imagery.

3.3.3 Study Population and Sampling:

A study population consists of all the elements from which the sample is actually selected. It is a well defined set of elements that have certain properties like that of soil/alluvial soils and rock samples. The target populations were all soils and rocks from the three study cites within the local areas. The researcher must therefore

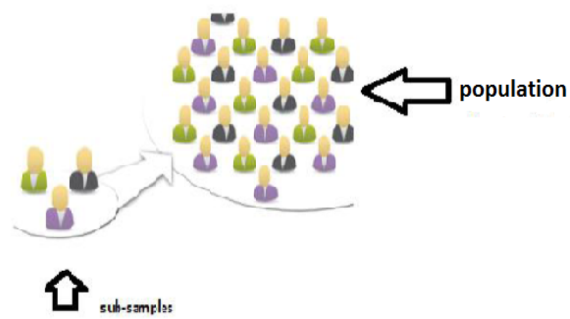

Figure 3.2: Sample collection technique

take the sample from the population (entire set of objects) because: the size of the population may not be too large to handle. The selection of sampling site, sampling method, and geological samples are dependent on (Chung et al., 2004) the aims of the research and the elements to be analyzed.

3.3.4 Sampling and Sampling Technique:

The major and most important component of this research program is sampling, which may be straightforward for existing surface rocks and soils, but challenging in underground development situations. Inappropriate or inconsistent sampling procedures produces unreliable data and can lead to erroneous interpretations.

Sampling is the process of selecting subjects from a designated population to represent the whole population (Wood & Haber, 2002). Probability sampling method was used for this study. In this method every member or element has a chance of being selected to participate in the study.

3.3.5 Sample Collection and prepation:

Soil/rock samples were collected during October 2010 from three different locations/research sites in East Gojjam. With a pair of gloves, the soil samples were collected from the surface to about 15 or 20 cm depth with hand auger and plastic shovel and transfered into pre−cleaned polyethylene bags. After taking each sample, the plastic shovel and the auger were washed with clean water and the hand gloves replaced to avoid contaminantions. Great care must be taken into account not to introduce contaminants that affect the samples to be irradiated such as sodium, chlorine and others.

The main aim in sampling is that the sample be truly representative of the soil in the area being sampled. Soil sampling can be done for diagnostic or troubleshooting reasons, where poor crop or pasture growth is noted in one area. This should allow more reasonable estimate to be made of soil test values with time.

The same procedure was followed when collecting the rock samples. All the samples were doubly bagged and boxed for safe transport from the field to the laboratory. At the laboratory, the soil/or rock samples brought from the field were spread on pre−cleaned polyethylene sheets for<gloved-hand-picking>of stones and plant materials.

The samples were stored (Choudhury et al., 2006) in pre−cleaned polyethylene capped bottles and handled with extreme care until the moisture is completely removed from the samples' surface. Just after removing the stones and vegetation, samples (Akhtar et al., 2005) were dried up at room temperature, sieved, placed in the plastic container and left for 30 days to remove moisture and water content. Dried samples were pulverized (Akhtar et al., 2005; Srinivas, 1995) into powder form, homogenized, weighed and placed in a plastic container for measurement.

The powdered samples were re−grinded again using local grinding machine, homogenized and packed in plastic containers, weighed, wrapped in polyethylene films. Finally, a refinement was done using portable mortar with pestle and then the homogenized samples were weighed, packed and sealed tightly with cup−holder.

The empty sample containers were first washed with tap water, dried, weighed and filled with soil/rock samples and weighed again. To do such activities clean instruments such as knife, file, tweezers, etc., and clean plastic gloves are needed. The containers were closed by plastic tapes wrapped over the caps. Sample procedurs were applied for the reference materials. The samples and standards were irradiated to more than 30 days to achieve secular equilibrium.

Samples and standards were bundled and sandwiched together and irradiated at a thermal flux (Dias & Prudêncio, 2007). Because of the low detection limits (Nassef et al., 2008) for many elements, sample collection and preparation is critical. Virtually any solid material can be analyzed by this method, but there are geometric constraints: the sample must fit into the irradiation vial (Alamoudi et al., 2006) and should present a consistent geometry. Before selecting material for analysis, consideration should be given to the geometrical factors.

3.3.6 Sample/Standard Mass:

All masses were measured using "Electrical Standard Balance". Samples and sample standards (Daia et al., 2001) are irradiated simultaneously and later acquired under the same counting conditions. The concentration of the elements of interest is calculated by comparison of the measured activity between the samples and standards. The mass of the sample could be of the order of 0.0001 gm, with concentrations of tenth of ppm or even smaller.

The sample holder: To center the sample in front of the neutron source, it can be hold in a proper position by using materials that neither absorb nor scatter the neutron flux.

3.3.7 Instruments of Data Collection:

The software, Genie−2000, is the research instrument/tool for collecting the required data from MCA (multi−channel analyzer) in order to realize the objectives of the research. The manner in which data is collected depends on the type and purpose of the research. In Nuclear Experiments, it would be asked to study gamma spectra using a few different functions of the computer software.

There are many commercial packages available which can calculate the statistics required for analysis of experiments. However, few are directed specifically towards experimental design. Most tend to concentrate on producing statistics, which are only realy relevant to passively accumulated data.

3.3.8 Data Analysis Procedure:

Data analysis is the process of looking at and summarizing data with the intent to extract useful information and develop conclusions. It also seeks to explain how different variables shape events and how they cause outcomes. Data analysis consists of examining, categorizing, graphing, tabulating, testing or otherwise recombining evidences to address the objective of the study.

3.4 Variable:

The research variables of any scientific experiment or research process are factors that can be manipulated and measured (Shuttleworth & Martyn, 2008). The variables of this research are "Elemental Analysis of Some Geological Samples and Isotopic Neutron Source".

Variability occurs when a single number is used to describe something that actually has multiple or variable values to adverse effects, or something that varies over time such as the population of an area. Variability occurs as a result of differences between the characteristics of different people or populations.

3.4.1 Independent Variable:

Isotopic neutron source is the circumstance or characteristics which the researcher can manipulate in his effort to determine what their connection with the observed phenomenon is. The independent variable is the variable which the researcher would like to measure (the cause). The researcher may actively manipulate the independent variable and see what effects has upon the dependent variable, or passively observe thereby collecting a series of measurements.

3.4.2 Dependent Variable:

Elemental analysis of geological samples is the circumstance or characteristics that changes, disappears or appears when the independent variable is manipulated. The dependent variable is also called the effect, response or outcome variable, at which the measurements are taken before and/or after manipulating the independent (also called the treatment or intervention) variable and depend on the independent variable.

3.5 Internal and External Validity:

Validity is the ability of the research instrument to measure what it is supposed to measure, like isotopic neutron source for activation purpose and HPGe−detector for detection purpose. All the instruments were checked by the research supervisor for the adjustement of efficiency and energy calibrations. In this study, the instruments were presented to the research supervisor for checking and relevant corrections were made. The idea of validity provides us with a unifying theory for understanding the consistency of good research.

3.5.1 i.Internal Validity:

Internal validity is the result of the study in true reflection of reality rather than the results of extraneous variables. Internal validity, or creditability, refers to the establishment of a phenomenon in a credible way.

3.5.2 ii.External Validity:

External validity is the ability of the results to be generalized beyond the sample used in the study. External validity, or transferability, refers to examining how applicable the research findings are to another setting or group (Shuttleworth & Martyn, 2009a).

3.5.3 Methods to Ensure Validity:

Validity concerns the accuracy of the sample irradiated, the data collected and the explanation offered. Generally it relates to the data and the analysis used in the research.The researcher ensured that the data collected was valid by consulting books, journals, periodicals, and information from the internet relevant to the aims and research questions of the research.

3.5.4 Methods to Ensure Reliability:

Reliability is the ability of the accuracy and consistency of the research instruments, which is obtained when the instruments are used again under the same conditions (Brink, 2006). Reliability tells us about a researcher's dependability and consistency throughout the research process: the degree to which it can be repeated. To ensure reliability, a researcher should make certain that if the same methods are used by different researchers and/or at different times, they should still produce similar results.

3.5.5 Practicality and Accuracy:

1. **Economy**: Some tradeoff ideal research project and the budget

2. **Convenience**: Easy to administer, easy to complete sample collection and preparation correctly

3. **Interpretability**: Relevant when person other than the test designer must interpret the result

The accuracy (Nimick et al., 1991; Jayaseelan et al., 2010; Leinweber et al., 2000) of each analytical technique has been checked by carrying out replicate analysis of standard materials having certified values.

3.5.6 Ethical Considerations:

Ethical guidelines serve as standards and the bases upon which each researcher ought to evaluate the research conduct. Ethics should be applied on all stages of the research work: planning, conducting and evaluating (Experiment−Resources, 2008a). In conducting this research work, soil/alluvial soils and rocks samples are collected especially in the research areas/cites. Permission might be required to collect the samples and to use the information provided in this research work if it is deemed essential. There are also ethical factors involved in conducting desk−based research. Theses include plagiarism and copyright issues. Citation is compulsary for accurate uses of any other research works.

3.6 Summary:

All research projects have the same philosophical foundations. These philosophical foundations provide justifications. When scholars engage in a research project, there are always underlying assumptions as to what entities exist what is called ontological assumptions, what research designs are appropriate for generating new knowledge known as methodological assumptions, and what criteria are appropriate for evaluating knowledge claims or epistemological assumptions.

The proposed method is found to be simple, accurate, precise and economical (Hirak et al., 2010) and can be employed for routine analysis.

Chapter 4

Experimental Work: Results and discussions

The first three chapters supplied backgound to the work done in “Elemental Analysis of Some Geological Samples” to internalize the princicples of “Neutron Activation Techniques”. These consecutive four chapters would describe the scientific experiments which are the main part of the work done for this research work. They would give extensive overviews of the work done on Neutron Activation Technique. All experiments were performed with isotopic neutron source and were mainly becoming familiar with the techniques of Neutron Activation Analysis.

The setup was thoroughly characterized and the possibilities and limitations of the technique were explored. Subsequent chapters aimed to present the results of the empirical research and elucidates the meanings and significances thereof within the current body of knowledge.

The procedure generally used for obtaining results from NAA was to obtain the isotopic neutron source facility of choice and the type of sample container that fits their particular reaction vessel. Generally heat sealant polyethylene vials were used. These could be generally made of plastics, and were selected based upon the size of the sample to be irradiated.

4.1 I.Elemental Analysis of Alluvial Soils Using NAA in Blue Nile River

4.1.1 Background:

Studies of different radionuclide and trace elements in the environmental samples is very important for health physics, research and education. Many forms of elements are possible in environmental samples which may be hazardous for humanitarian and also animals. Most of them have the potential for both beneficial and harmful effects.

Alluvial soil samples were analyzed to provide information on the elemental composition. The concentrations of various elements, in alluvial soil samples collected from Blue Nile River Basin in the direction of water flow, were determined using Neutron Activation Analysis (NAA).

4.1.2 Introduction:

The study areas are located in North−western side of Ethiopia: Blue Nile River basin ($10^0 04'02"N$) latitude and ($38^0 11'24"E$) longitude with an elevation of 1036 meter above sea level (asl). The Blue Nile and its tributaries rise on the Ethiopian plateau (Sutcliffe & Parks, 1999; Dereje et al., 2002; Gani et al., 2008; Beaulieu & Gaonaćh, 2002), which is concentrated at elevations of (2000−3000) meter, with several peaks upto 4000 meter or more. The plateau country (Dereje et al., 2002; Gani et al., 2008) is not flat but very broken and hilly, with grassy uplands, swampy valleys, scattered trees and bushes. There are occasional rocky peaks, some of which are of volcanic origin (Hagos et al., 2010).

The curious course of the river may follow the original drainage pattern radiating from such volcanic centers. The basin is cut by deep ravines or canyons (Ayalew & Yamagishi, 2003) in which the Blue Nile and other rivers flow. The valley of the Blue Nile is 1300 meter deep in place and the course of the river is difficult to cross. The sample locations (see Maliszewska−Kordybach et al., 2008; Angelal et al., 2010; Kijowska, 2004, chap. 3) that were determined by the use of GPS were plotted on the geological map of the study area fig.(4.1). Alluvial soils were collected from different

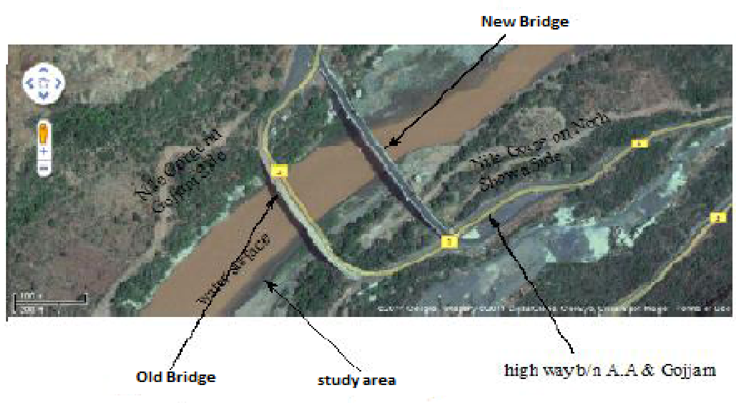

Figure 4.1: Alluvial soil taken under Blue Nile bridge

positions of Blue Nile River basin (Steenhuis et al., 2009) in the direction of water flow. The sample locations were recorded with a GPS (Global Positioning System) and it is ($10^0 04' 27" N$) latitude and ($38^0 11' 24" E$) longitude.

An alluvial soil sample and two standards of cupric sulphate and arsenic trioxide were inserted into the (Daraban et al., 2005) isotopic neuton source of (Am^{241}- Be^9) type (irradiation block) (Filby, 1995; Karadag et al., 2003; Sadiq et al., 2010) and irradiated (Ljubenov & Milosevic, 2004) for about 12 days. The measurements were repeated using highly pure potassium iodide samples (using I^{128}, 25 min activity).

Detector efficiency :

In order to perform a delayed gamma neutron activation analysis (DGNAA) measurement it is important to know the counting efficiency of the detector as a function of energy. It represents the number of recorded pulses under the photopeak devided by the number of the gamma−rays emitted by the source. The counting efficiency depends on the gamma−ray energy but it is also strictly connected to the (Stella, 2011; Firestone & Trkov, 2005) germanium geometry: <distance-detector-source>, presence of shilding. It can be evaluated experimentally by the use of a gamma source with known disintegration rate, applying the formula (sea chapter two):

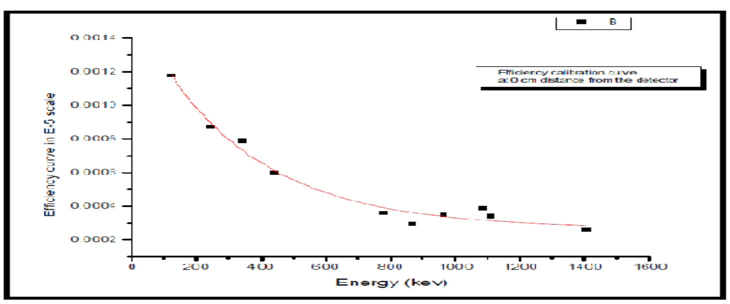

Figure 4.2: Efficiency Calibration at 0 cm

4.2 Experimental measurements performed at NAA:

Experiments were performed to evaluate elemental contents and concentrations of geological samples at the sound level. The samples had been previousely packed in bages, mounted and located alwayes at the sample position. For each sample the net area under the <full-energy-peak>was determined subtracting from the gross area of a trapezoidal baseline evaluated by averaging the left and right channels using Genie−2000 software program. The unknown samples and standards (irradiated together in an isotopic neutron source) are sequentially placed infront of the detector for which the photo−peaks of the standards are depicted in fig.(4.3 and 4.4).

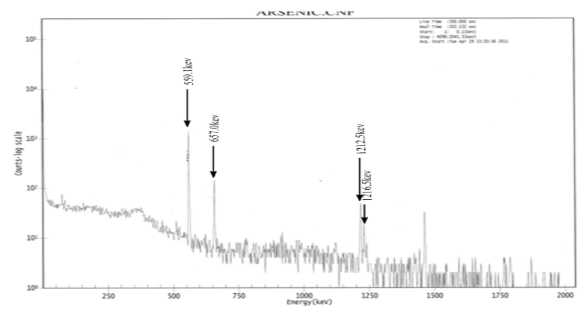

Figure 4.3: Analysis of gamma spectra for Arsenic trioxide

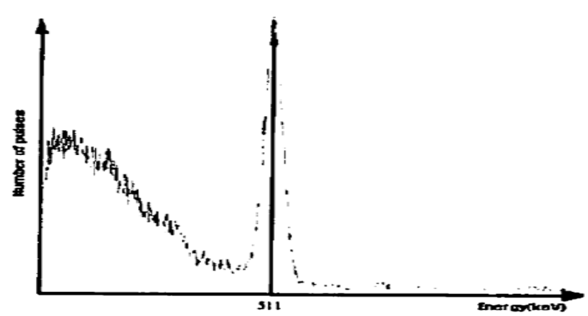

Figure 4.4: Copper-64 spectrum

It has been noticed that the appearance of 511 kev annihilation peak, which shows that the sample contains the Cu^{64} radionuclide.

The decay scheme of copper is: Cu^{64} disintegrates by beta minus emission to Zn^{64}

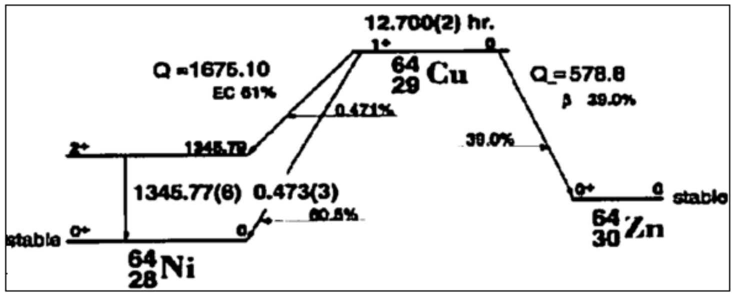

Figure 4.5: Decay Scheme of Cu^{64}

ground state and by electron capture to the excited level and the groundstate of Ni^{64}.

The isotope produced in the experiment is I^{128} which is produced by neutron capture of I^{127}, and has a half−life of 25.0 min. This isotope decays by both beta decays and electron capture as shown: Two standards are placed on both sides of the sample for

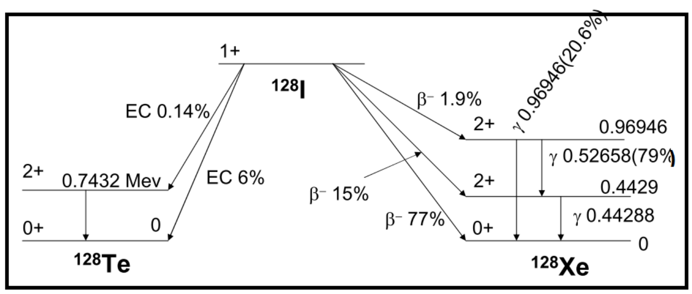

Figure 4.6: The decay of I^{128}

activation purpose.

4.2.1 Qualitative Analysis of NAA experiment:

Background optimization: The background spectrum repesents the signal that is not related to the sample: it is desirable to reduce this contribution as much as possible becuase it negatively affects the detection limit of the measurement system. Background components can be distinguished:

the room−background, due to the γ−naturally present in the room in the absence of the sample. To perform the comparison, the room background spectrum was subtracted from the experimental one.

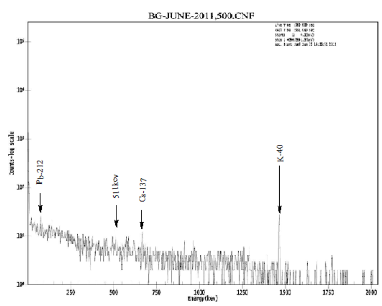

Figure 4.7: Analysis of Gamma Spectra for Room Background

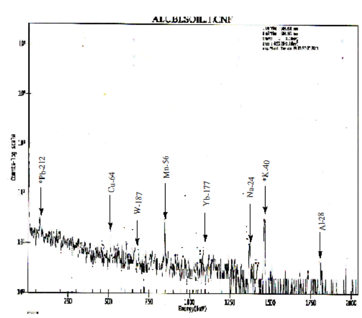

Figure 4.8: Analysis of Gamma Spectra for Alluvial Soil

$*K^{40}$ and $*Pb^{212}$ are natural peaks and other peaks are not shown on the spectrum too.

The area beneath these peaks is integrated, typically using Genie−2000 software code, for both samples and standards, and this area is proportional to isotope abundance.

Decay Scheme:

For the irradiated Na, the following energies were found in (kev): (996.09, 1368.7, 2754.2, 2869.7, 3866.5, 4238.4) which it was attributed as belonging to Na^{24} and for which the disintegration scheme is illustrated in fig.(4.9). The transitions measured in this work are denoted by heavy lines.

Decay scheme of sodium: Na^{24} disintegrates by emission of beta−minus particles

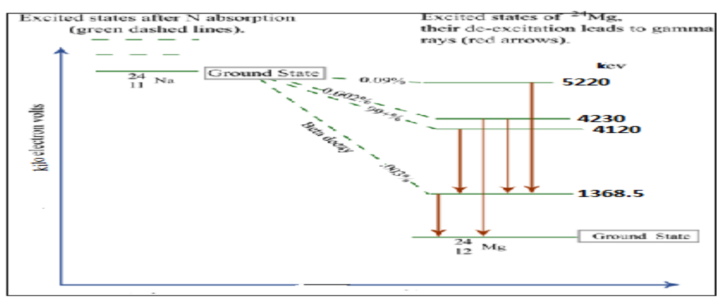

Figure 4.9: Nuclear energy levels diagram for Mg^{24} showing the excited states populated during beta decay of Na^{24}

(100%). This process is followed by two gamma−rays in a cascade (2754 and 1393 kev) which leads through the 1368 kev level to the ground state of Mg^{24}. Most compounds contain sodium, and when it is exposed to neutron, it will capture them to form the radioisotope Na^{24}. Due to the high transition energies internal pair formation takes place.

For Mn, the following energies were found in (kev): (846.8, 1810.8, 2113.2, 2522.88) which it was attributed as belonging to Mn^{56} and for which the disintegration scheme is depicted in fig.(4.10).

Decay scheme of Manganese: Mn^{56} decays by beta minus emission to excited levels

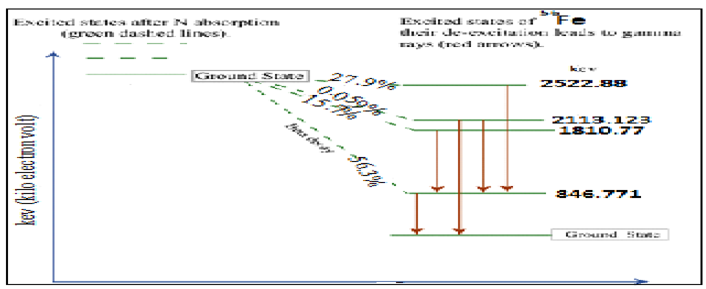

Figure 4.10: Nuclear energy levels diagram for Fe^{56} showing the excited states populated during beta decay of Mn^{56}

of Fe^{56}. The transitions measured in this work are denoted by heavy lines. A search was made for other possible gamma−rays with energies below 1500 kev. An upper limit of 0.0004 times the 846.8 kev gamma−ray intensity can be placed on the intensity value for the 299 kev gamma−ray which might proceed from the fourth to the third excited state.

Radioisotopes (Diago, 2008) that decay with the emission of gamma radiation are much easier to detect using the germanium detector. Becuase the energy resolution of germanium detector is much better than any other detectors. Ir^{194} decays 100%

by beta minus to Pt^{194}. Most of the decay populates to the ground state. All the

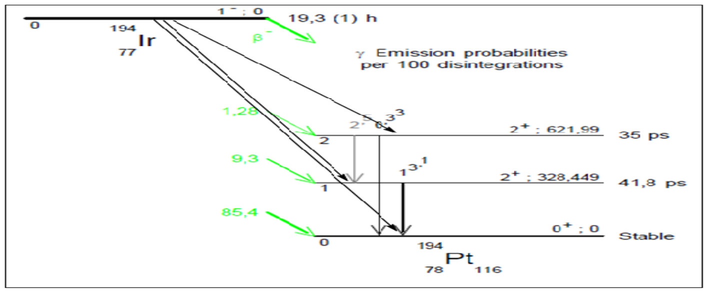

Figure 4.11: Decay Scheme of Ir^{194}

figures show the decay schemes of the irradiated isotopes by (Daraban et al., 2005) isotopic neutron source. The amount of produced daughter nuclei were estimated by measuring the gamma−rays they emitted. The yields of the gamma−rays emitted from the irradiated target were measured using a high purity germanium detector. The transitions measured in this work are denoted by heavy lines and the energies of the levels are obtained from present measurements.

Half-life and decay constant determination:

If N is the number of radioactive nuclei, the decrease in N with time "t" is given by the first order rate law:

$$\lambda = \frac{-(dN/dt)}{N} \tag{4.2.1}$$

in which λ is a decay constant regardless of the age of the atom.

After some mathematical manipulations:

$$N = N_o e^{-\lambda t} \tag{4.2.2}$$

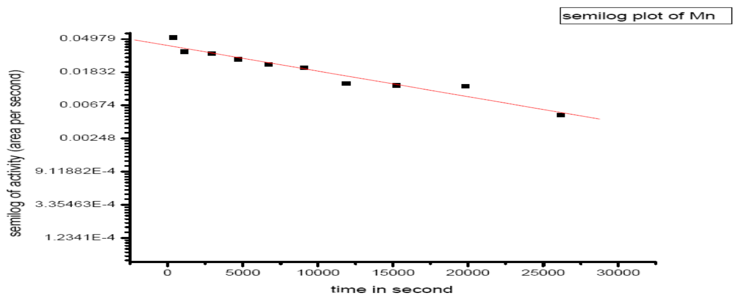

Figure 4.12: The exponential decay of activity of Manganese in semilog plot

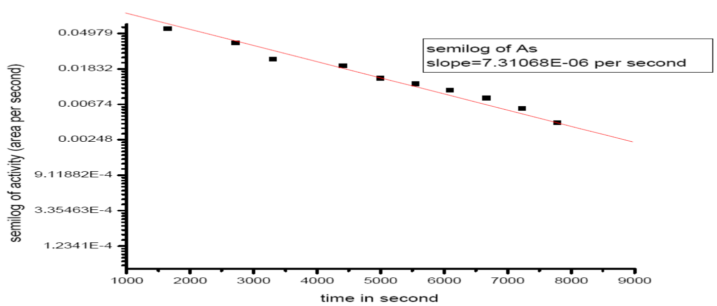

Figure 4.13: The exponential decay of activity of Arsenic in semilog plot

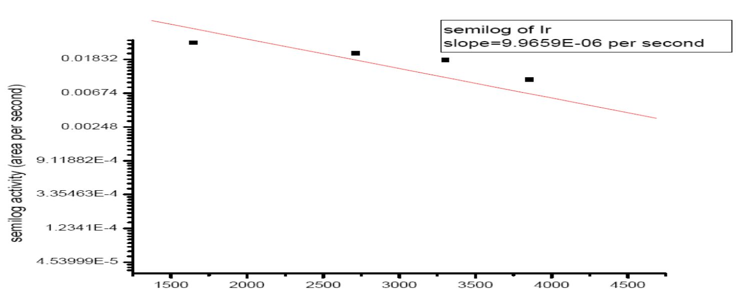

Figure 4.14: The exponential decay of activity of Ir in semilog plot

This is the decay equation showing the exponential decrease in the N with time and the half−life can be calculated as:

$$t_{1/2} = \frac{\ln 2}{\lambda} \tag{4.2.3}$$

Similar half−life was calculated for the confirmation of all the elements in the conducting experiment.

4.2.2 Quantitative Analysis of NAA:

Elemental identification and Quantification:

The evaluation of the different nuclides contained in a sample can be performed by the gamma−ray full energy peak identifications. The peak identification is performed by comparing the obtained spectrum with known delayed gamma−ray data.

Experimental Elemental Concentrations:

The (NAA) technique has led to a successful determination of twelve elements in the alluvial soil sample around the research area. Based on the energies obtained in the spectrum as well as on the disintegration schemes/curves, the possible identification of radioisotopes and their concentrations gave values of (5.5 ppm for Mn^{56}); (52.5 ppm for Na^{24}); (939.5 ppm for Al^{28}); (21.5 ppm for W^{187}); (96.5 ppm for Cu^{64}); (7.5 ppm for As^{76}); (1497.5 ppm for Ru^{105}); (31593.5 ppm for Cd^{117}); (770.5 ppm for Yb^{177}); (1 ppm for Ir^{194}); (0.06 ppm for Eu^{152}) and (187.5 ppm for Ba^{139}).

This serves as the basis to compare the elemental composition of the raw material (alluvial soil) with what is obtainable in the synthesized sample. Calculations of count rates for each detected photo−peaks and concentrations (activity per unit mass or specific activity) of the detected radionuclide depend on the establishment of secular equilibrium in the sample. The elemental concentrations in alluvial soil sample by using (NAA) are shown in the table (4.1). The energy of the gamma−rays, their

Table 4.1: Elements obtained from alluvial soil collected from the territories of Blue Nile basin using Neutron activation analysis (Irradiated for 12 days with known standards: Arsenic Tri−oxide and Cupric sulphate)

The radio nuclide	I_γ in(%)	The Half life	Concentration in (ppm)	E_γ in (kev)	f_γ in (%)
Mn^{56}	100	2.56h	0.0057/0.0065	846.8	99
Na^{24}	100	14.90h	0.0493/0.0563	1368.6	100
Al^{28}	100	2.24m	0.7956/0.1084	1778.9	100
W^{187}	28.4	23.72h	0.020598/0.02353	685.7	26
Cu^{64}	69.1	12.7h	0.0997/0.0940	511	37
As^{76}	100	26.4h	0.0077/0.0089	559.9h	45
Ru^{105}	18.0	4.44h	1.6629/1.3339	468.6	17
Cd^{117}	7.6	3.36h	29.4739/33.7141	1066.8	23
Yb^{177}	12.73	1.90h	0.6725/0.7693	1080.2	5
Ir^{194}	61.5	19.28h	0.0014/0.0013	688	59
Eu^{152}	47.77	9.3h	0.00005/0.00006	963	43
Ba^{139}	71.66	83.06m	0.1752/0.2004	1420.2	22

intensity and half−lives of various isotopes were taken from table of isotopes (Chu et al., 1998) and the values of thermal neutron capture cross−sections were taken from (IAEA, 1987).

Table (4.2) shows that major, minor and trace elements.

Table 4.2: Concentration of major, minor and trace elements)

Stable isotope	I_γ (%)	Concentration(%)		
		Major	**Minor**	**Trace**
Cd^{116}	7.49	2.9/3.4		
Eu^{151}	47.81		0.5/0.5	
Ru^{104}	100		0.17/0.13	
Ba^{138}	71.698		0.018/0.02	
Yb^{176}	31.83		0.067/0.077	
Al^{27}	100		0.1/0.11	
Cu^{63}	69.1		0.01/0.01	
As^{75}	100			0.00077/0.00088
Ir^{193}	62.7			0.00014/0.00013
Mn^{55}	100			0.0005/0.00065
Na^{23}	100			0.005/0.0056
W^{186}	28.43			0.002/0.002

4.3 Results and Discussion:

Experimental results include Cd^{117} which constitutes as major element; Ru^{105}, Al^{28}, Yb^{177}, Ba^{139} and Cu^{64} which constitute as minor elements and W^{187}, Na^{24}, Ir^{194}, As^{76}, which constitute as trace elements in alluvial soil and were determined to be above detection limit. The detection limit for Mn^{56} and Eu^{152} is in good agreement (James & Cynthia, 2002) to the mass calculated after activation.

The (NAA) technique has lead to a successful determination of twelve elements in the alluvial soil around the research area. These elements include heavy metals like Yb^{177}, Mn^{56}, Cu^{64}, Ru^{105}, Na^{24}, and As^{76} among others. Some of these metals have been known to be toxic (Leszczynska & Ahmad, 2006) and dangerous to human health specially when they are found in water and growing crops (Putaraporn et al., 1995).

Arsenic, (IAEA, 1997; Wei & Zhang, 2007; Leszczynska & Ahmad, 2006) which is a semi−metallic element, is known to be strongly poisonous. The presence of this element in the soil posses a serious threat to human health, especially when it is presented in edible crops, through their roots.

Manganese (IAEA, 1997) poisoning has been linked to impaired motor skills and cognitive disorders. The human body contains about 10 gm of manganese which is stored mainly in the liver and kidneys. In the human brain, the manganese is found as manganese metalloproteinase most notably glutamine syntheses in astricytes. Elemental analysis of alluvial soil samples has been important in identifying the effects of such elements on the environment and health.

4.3.1 Conclusion:

The area, exposed to wind, close to running river and situated on a sloppy agricultural farm land, encourages erosion/soil movement (Havstad et al., 2006; Ritchie et al., 2003) by wind and water (Councile, 1994; Konz et al., 2010) and also increases the chance of leaching. This is the reason for having most of the elements in very low amount. All samples taken from the surface layer $(0 - 30)$ cm of alluvial soils (Zhao et al., 2011) are widely formed from fertile agricultural lands (mostly farm fields).

No internationally agreed system for classification of the soil based on the grain size (American−Socity & for−Testing-and Materials, 1972), it is however very useful to perform a simple test to identify the range of particles covered in the soil matrix, that are visible to the naked eye. The soil characterization has been done using

Non−destructive Neutron Activation Techniques combined with high−resolution germanium γ−ray spectrometry, a powerful and effective (Win, 2004; Abd et al., 2009; Sarathi et al., 2008) instrumental multi−element analytical technique, for analyzing major, minor and trace elements in soil.

Chapter 5

II.Elemental Analysis of soil samples from farmlands of Yebrage Hawariat using NAA:

5.1 Background

A combination of continuous cereal cropping, tillage and stubble removal reduces soil fertility and increases soil erosion on sloping land. This study is used to focus on the investigation and explanation of changes in soil fertility under stubble removal and retention in farmlands of Yebrage where soil is prone to sever erosion.

Neutron activation Analysis (NAA) is used to determine the elemental composition present in soils collected from farmlands of Yebrage near Chemoga River Basin (Sutcliffe & Parks, 1999). The macro/micronutrient and organic matter deficiencies have been verified in many soils through increased use of soil testing and plant analysis.

It will be concluded that stubble retention should be conducted to increase crop productivity, improve soil fertility as well as agriculture attainability in agricultural land plot of Yebrage.

5.2 Introduction

Farmland owners are continually searching for environmentally acceptable and economically viable means of sewage sludge (Richards et al., 2000) disposal. The environmental (Kadi, 2009) protection agency promotes the recycling of sludge material on some crop lands since it is an excellent source for several plant nutrients (Spaccini et al., 2006). Crop stable is a main agricultural waste material as well as a renewable resource, due to being rich in nitrogen (N), phosphorus (P) and potassium (K).

Farmers in Yebrage (Senishaw, 2003) near Chemoga River (Teferi et al., 2010) had a long tradition of efficient recycling of organic residues in agriculture, but this tradition is rapidly disappearing following the intensification of agricultural production, the increased use of inorganic fertilizers, and the increasing urbanization and decoupling of crop and animal productions. The intensification of agricultural production has greatly increased the agricultural production, but at the same time, it has contributed to a decrease in response use of efficiency, land degradation through increased wind and water erosion and pollution of ground and surface water. Agriculture, the main economic activity and a soil based industry practiced in Yebrage, extracts nutrients (Berry et al., 2007) from the soil.

It has been well established that optimum yields generally are not possible without primary and secondary nutrients and also organic matter. The macro/micronutrient and organic matter deficiencies have been verified in many soils through increased use of (American−Socity & for−Testing-and Materials, 1972) soil testing and plant analysis. Soil organic matter (SOM) (Sleutel et al., 2010) is that fraction of the soil composed of plant and animal remains in various stages of decomposition and synthesis.

Carbon (C), Hydrogen (H) and Oxygen (O) are obtained from the atmosphere and from the water around the plant. The other macronutrients are Nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S) which should be available from the plant uptake from the soil. Nitrogen (N) plays (Nathalia et al., 2011) an important role in agricultural productivity (Abebe, 2005). The micronutrients obtained from the soil are boron (B), Nickel (Ni), copper (Cu), chlorine (Cl), iron (Fe), manganese (Mn), molybdenum (Mo), zinc (Zn), and cobalt (Co) which are very essential because the absence of any one of these will cause the plant to grow poorly or develop disease.

The limiting conditions of nutrients (Spaccini et al., 2006) in soils are vitally important for crop growing because it is well known that such crops need to extract high quantities of nutrients from the soil. This explains the favorable responses to fertilization, given that major yields result from high nutrient input. Soil sampling and testing provided an estimate of the capacity of the soil to supply adequate nutrients to meet the needs of growing crops. Soil degradation (Department-Agriculture, 2008) results the reduction of soil in quality and quantity by the agents of soil erosion/soil movement.

Soil (Ritchie et al., 2009) is a natural resource that is frequently mistreated. Life is dependent on soil as much as it is on water and air. The NAA is accepted as an important technique (Santoso et al., 2010) for elemental analysis of farmland soil samples. The basic essentials required to carry out an analysis of soil samples by neutron activation analysis are a source of neutron, $HPGE$ gamma$-$ray spectrometer coupled with a MCA and printer.

5.3 Nuclear Experimentation

The study areas are located in North$-$western side of Ethiopia: Yebrage ($10^0 20' 02" N$) latitude and($37^0 43' 47" E$) longitude with an elevation of "2450" meter above sea level (see Maliszewska$-$Kordybach et al., 2008; Angelal et al., 2010; Kijowska, 2004, chap. 3). To investigate the impacts caused by soil erosion of the tillage around the farmland of Yebrage and its surroundings in East Gojjam, soil samples (Harb et al., 2008) were collected from different farmlands of "Yebrage" near Chemoga River (Teferi et al., 2010) and analyzed. A composite sample consisting of three sub$-$samples was collected with a minimum distance of 30 m between sub$-$samples. Each sampling point was geo$-$referenced by Global Positioning System (GPS) (sea chapter two).

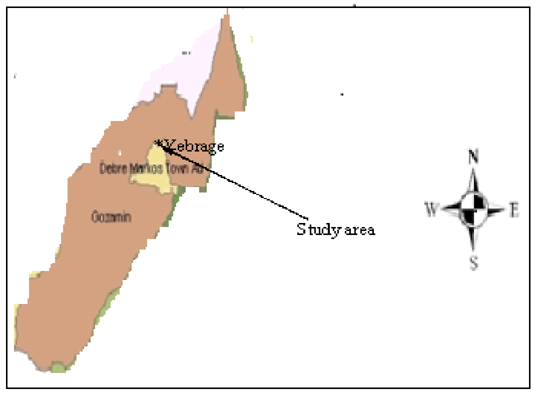

Figure 5.1: The location and topography of the study area

5.4 Experimental Measurements performed at NAA:

Background Optimization: (sea chapter four).

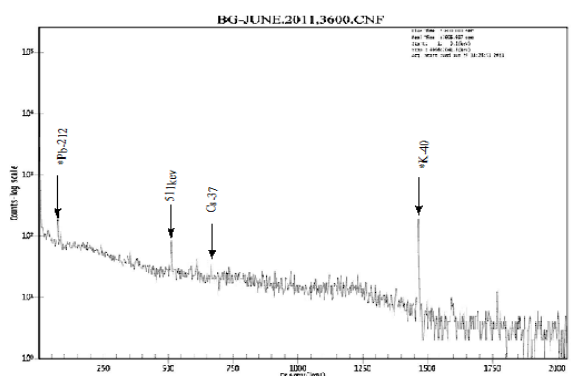

Figure 5.2: Analysis of Gamma Spectra for Room Background

*The gamma−rays are analyzed qualitatively only since they are naturally occurring radioisotopes. The decay scheme gives energy difference/Q−value.

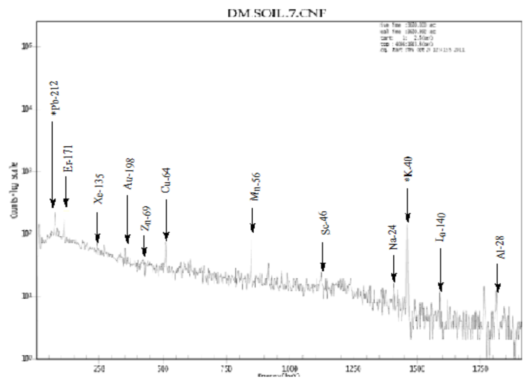

Figure 5.3: Analysis of Gamma spectra for farmland soil of Yebrage

Decay schemes: For Sc the following energies were found in (kev): (889.3, 1120.6, 2009.8) which it was attributed as belonging to Sc^{46} and for which the disintegration scheme is illustrated in fig.(5.4). The transitions measured in this work are denoted by heavy lines and the energies of the levels are obtained from present measurements.

Sc^{46} disintegrates by 100% beta minus emission to excited level in Ti^{46}. The main path leads to 2009.8 kev level of Ti^{46}. The ground state of Ti^{46} is reached via a gamma cascade of 1120.5 and 889.3 kev.

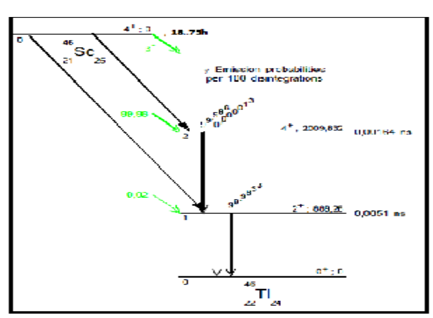

Figure 5.4: Decay Scheme of Sc-46

In fig.(5.5), Mo^{99} disintegrates to the Tc^{99} excited levels by beta minus emissions. The 142 kev excited level (Tc^{99m}) has a half−life of 6.0067 h. At (t>60 h), both Mo^{99} and Tc^{99m} are considered to be in equilibrium.

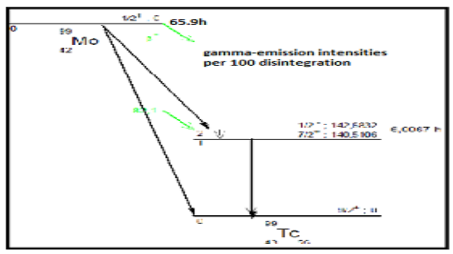

Figure 5.5: Decay Scheme of Mo-99

Half-life determination and element identification: Sample decay curve:

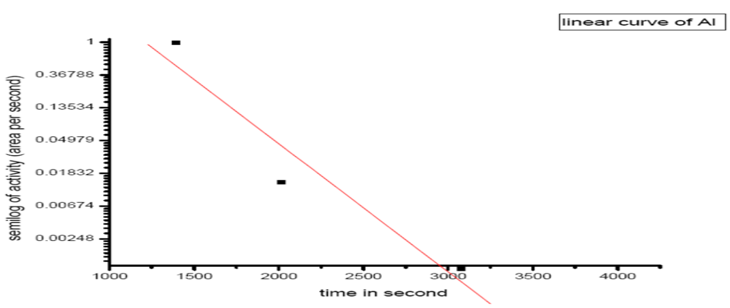

Figure 5.6: The exponential decay of activity of Al in semilog plot

Using the same expressions of equations (4.2.1) and (4.2.3), the decay constants and half−lives of the curves can be calculated (sea chapter four).

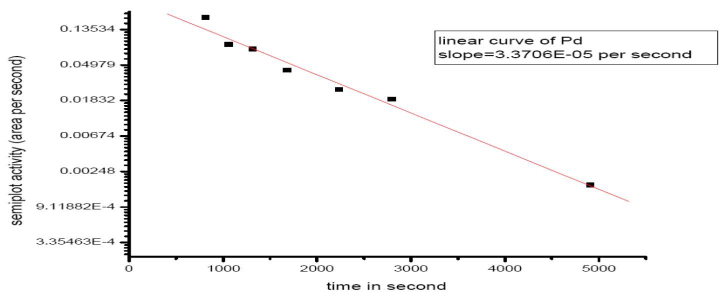

Figure 5.7: The exponential decay of activity of Pd in semilog plot

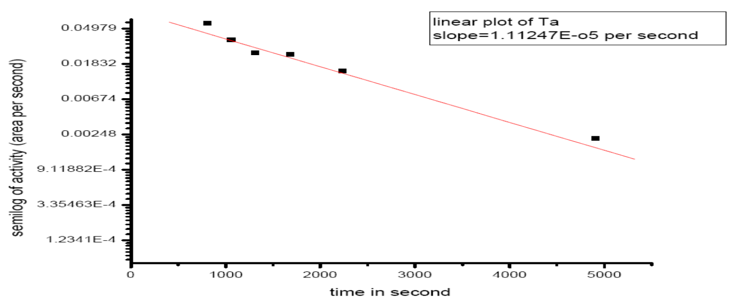

Figure 5.8: The exponential decay of activity of Ta in semilog plot

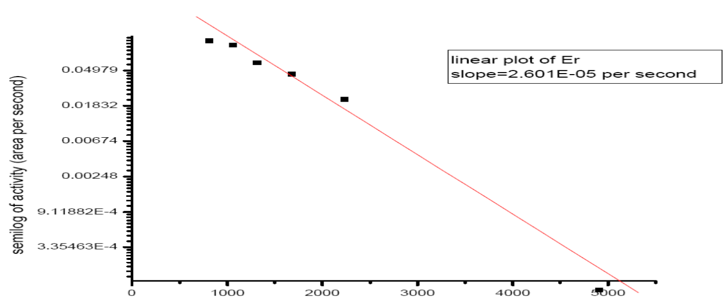

Figure 5.9: The exponential decay of activity of Er in semilog plot

5.4.1 Elemental identification and Quantification:

Activity concentration:

The (NAA) technique has lead to a successful determination of fifteen elements in the soil sample from farmland of Yebrage. The results show that NAA of soil samples around the study area gave values of (3.54± 0.0006 ppm for Er^{171}), (0.034±0.00006 ppm for Mn^{56}), (6.5±0.0008 ppm for Pd^{111}), (0.49±0.0002 ppm for Cu^{64}), (0.22±0.0001 ppm for Ga^{72}), (27.14±0.005 ppm for Ta^{182}), (0.355±0.0002 ppm for Zn^{69}), (0.00023±0.0001 ppm for Au^{198}), (1.8±0.0004 ppm for Xe^{135}), (0.016±0.00004 ppm for Sc^{46}), (0.414±0.0006 ppm for Cd^{115}), (1.89 ±0.0004ppm for Mo^{99}), (18.798± 0.072 ppm for Al^{28}), (0.0403± 0.00335 for La^{140}) and(0.3252± 0.0095ppm for Na^{24}). This serves as the basis to compare the elemental composition of the raw material (soil) with what is obtainable in the synthesized sample.

Calculation of count rates for each detected photo−peak and radiological concentrations (activity per unit mass or specific activity) of detected radionuclide depend on the establishment of secular equilibrium in the sample. The element concentrations in agricultural soil sample by using (NAA) are shown in the table 5.1.

The energy of the gamma rays, their intensity and half lives of various isotopes were taken from table of isotopes (Chu et al., 1998) and the values of thermal neutron capture cross−sections were taken from (IAEA, 1987).

Table 5.2 shows that one as major element, nine as minor elements and four as trace elements.

Table 5.1: Elements obtained from soil collected from Farmlands of Yebrage using Neutron activation analysis (Irradiated for many days with known standards: Potassium Iodide)

The radio nuclide	Natural Abundance in(%)	The Half life	Concentration in (ppm)	Gamma energy in (kev)	Gamma abundance in (%)
Er^{171}	14.88	7.52h	3.54±0.0006	112	21
Mn^{56}	100	2.56h	0.034±0.00006	846.7	99
Pd^{111}	34	5.5h	6.51±0.0008	171	20
Cu^{64}	69.1	12.7h	0.49±0.0002	511	37
Ga^{72}	40	14.10h	0.22±0.0001	629	25
Ta^{182}	100	16.5h	27.14±0.005	1123	35
Zn^{69}	18.6	13.76h	0.355±0.0002	440	100
Au^{198}	100	2.7h	0.0023±0.0001	351	95
Xe^{135}	10.4	9.14h	1.8±0.0004	248	90
Sc^{46}	100	18.75h	0.016±0.00004	1118	100
Cd^{115}	28.8	53.46h	0.414±0.0006	491	8
Mo^{99}	24.4	65.9h	1.89±0.0004	141	5
Al^{28}	100	2.24m	18.798±0.072	1778	100
Na^{24}	100	14.9h	0.3252±0.0095	1369	100
La^{140}	99.9	40.2h	0.0403±0.00335	1596	95

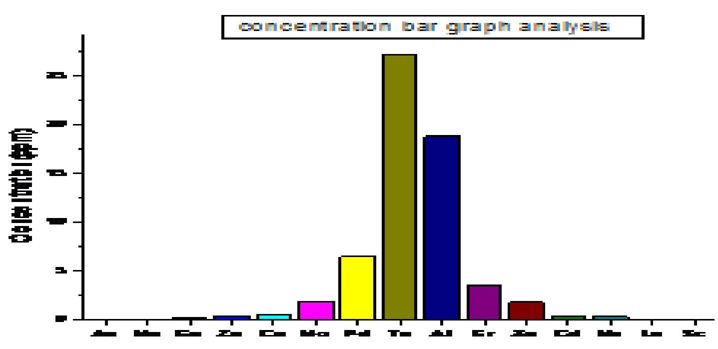

Figure 5.10: Graphical analysis of soil sample using bar graph

Table 5.2: Concentration of major, minor and trace

Stable isotope	I_γ (%)	Concentration(%)		
		Major	**Minor**	**Trace**
Ta^{181}	100	2.77		
Al^{27}	100	1.89		
Cd^{114}	28.8		0.042	
Er^{170}	14.88		0.595	
Pd^{110}	34		0.86	
Cu^{63}	69.1		0.049	
Ga^{71}	40		0.0219	
Zn^{68}	18.6		0.036	
Xe^{134}	10.4		0.19	
Mo^{98}	24.4		0.192	
Au^{197}	100			0.00023
Mn^{55}	100			0.00338
Sc^{45}	100			0.001579
Na^{23}	100			0.000896
La^{139}	99.9			0.0043

The Experimental elemental concentration: include Ta^{181} and Al^{27} which constitute as major elements; Cd^{114}, Er^{170}, Pd^{110}, Cu^{63}, Ga^{71}, Zn^{68}, Xe^{134}, and Mo^{98} which constitute as minor elements and, Au^{197}, Mn^{55}, Na^{23}, Sc^{45} and La^{139} which constitute as trace elements in soil. Zn^{68}, Cd^{114} were determined to be below detection limit, while Ta^{181}, Sc^{45}, Au^{197}, Mo^{98}, Ga^{71}, Er^{170}, La^{139} and P^{110} were determined above detection limit.

5.5 Results and Discussion:

The NAA technique has lead to a successful determination of twelve elements in the soil from farmland of "Yebrage", the research area. The elements determined include heavy metals like Er^{171}, Mn^{56}, Cu^{64}, Pd^{111}, Ga^{72}, Ta^{182}, Zn^{69}, Au^{198}, Sc^{46}, Cd^{115},

La^{140} and Mo^{99} among others.

But the soil lacks organic matter, macro and micronutrient, which are very important, that plants receive from the soil to survive. Carbon (C), hydrogen (H) and oxygen (O) are obtained in almost unlimited amounts from the atmosphere and from the water around the plants. The other macronutrients are nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg) and sulfur (S) which should be available from plant uptake from the soil. And the micronutrient obtained from the soil are boron (B), nickel (Ni), copper (Cu), chlorine (Cl), iron (Fe), manganese (Mn), molybdenum (Mo), $zinc(Zn)$ and cobalt (Co) which are very essential because the absence of any one of these will cause the plant to grow poorly or develop disease.

Manganese found here is an essential element for plant growth and plays a key role in photosynthesis and other critical pathways in the plant. Manganese can substitute for magnesium in some reactions in the plant. Manganese has been found to suppress take−all patch caused by Gaeumannomyces graminis on bent−grass. Manganese (IAEA, 1997) poisoning has been linked to impaired motor skills and cognitive disorders.

The human body contains about 10 gm of manganese which is stored mainly in the liver and kidneys. In the human brain, the manganese is found as manganese metalloproteinase most notably glutamine syntheses in astricytes. Toxicity of Mn can result in some acidic, high−Mn soils. Elemental analysis of agricultural soil samples has been important, because of the effects of these elements on the environment and health.

Sodium is present in the earth's crust at about the same concentration as potassium but most turf−grass plants have developed high selectivity for uptake of potassium. Sodium has been found to be an essential nutrient for a few plant species but in practical terms, sodium plays a detrimental role in turf−grass and soil management.

Sodium plays a direct role in turf−grass susceptibility to rapid blight caused by Labyrinthula terrestris. Increased sodium results in increased susceptibility to rapid blight. In addition, high salinity and sodium stress have been implicated in increasing susceptibility to senectotrophic pathogens that include anthracnose (Colletotrichum cerealis) and the leaf spots and melting out caused by Bipolaris spp. and Curvularia spp. In addition, low sodium soils benefit general plant health and aid in preventing weed invasion and recovery from damage caused by insects.

Palladium (Cavalcante & Saiki, 2007; Firestone et al., 2006) is quite similar in its characteristics as platinum, it is used in the semiconductor industry, in catalytic converters, in photography, jewelry manufacture, and in electronics. Gastro−intestinal absorption is limited, while exposure via inhalation is likely to result in more retentions of the element. Excretion via the urine and biliary tract is apparent although accumulation of the element can occur in kidney, spleen and liver.

Cadmium can adversely affect the heart, bone and tests. Smoking and high sugar diets appear to increase Cd levels. In children, elevated Cd has been correlated with lowered IQ. The concentration of Cd in foods is related to its level in the soil and its bio−availability. Crops cultivated in contaminated soil will present higher concentration of Cd than those from uncontaminated soil. Cadmium toxicity impacts the kidney, where damage to proximal tubules has been described. Also, cadmium compounds are classified as carcinogenic to humans. Potential sources include drinking wastewater, processed foods, cigarette smoke, paint pigments, and silver polish.

Most copper is concentrated in liver, brain, and hair but is present in all other tissue. Best dietary sources are whole grains, nuts, seeds, beans, liver and selfish. Loss of these biochemical functions can lead to anemia, neural degeneration, lung and bone problems.

Sources of zinc in the diet include whole grains, nuts, seeds, and seafood, especially shellfish. Growth and repair of any tissue is dependent on zinc as an activating cofactor for (DNA/RNA) (Stump et al., 2002) polymerase. For this reason, zinc is vital to the healing of wounds and skin disorders. There are many similarities between the immunologic effects of zinc deficiency and those of $AIDS$.

Mo is an essential trace element that is an activator of specific enzymes such as: xanthine oxidase (catalyzes formation of uric acid), sulfite oxidase (catalyzes oxidation of sulfite to sulfate), and aldehyde dehydrogenase (catalyzes oxidation of aldehydes). Possible effects or symptoms consistent with Mo deficiency are: subnormal uric acid in blood and urine, sensitivity or reactivity to sulfites, protein intolerance (specifically to sulfur−bearing amino acids), and sensitivity or reactivity to aldehydes. True

Mo deficiency is uncommon but may result from: a poor−quality diet, gastrointestinal dysfunctions, or tungsten exposure. Tungsten can be a powerful antagonist of Mo retention in the body. Copper overload can also reduce Mo retention. Because normal blood and blood cell of Mo levels are very low (a few parts per billion), blood measurement is not an appropriate tissue for confirmation of subnormal molybdenum. Confirmatory tests for Mo deficiency include measurement of urine sulfite concentration (increased in Mo deficiency), measurement of blood/urine uric acid level (decreased in Mo deficiency), and measurement of urinary Mo content.

Toxic metals may exchange between blood plasma and erythrocyte after a person is exposed. The concentration of these metals in erythrocytes also is determined the content of tissue where erythrocytes originate: the bone marrow. The bone marrow exchanges the metals with the mineral matrix of bone. The distribution of elements between bone and various soft tissues varies with each element. The potentially toxic elements (Leszczynska & Ahmad, 2006) vary considerably with respect to their relative toxicities. The accumulation of more than one of the most toxic elements may have synergistic adverse effects, even if the level of each individual element is not strikingly high. Toxic elements (Quarshie et al., 2011; Leszczynska & Ahmad, 2006) are associated with a number of different toxicological effects which begin to manifest when a particular toxic elements is present in the human body above a certain threshold.

5.5.1 Conclusion:

Samples are routinely tested for a variety of major, minor plant nutrients. Plants grown under optimum soil fertility levels typically are healthier and more vigorous. The soil covering the surface of the earth has taken millions of years to form and we must learn to respect it. This means that soil is a nonrenewable resource and once destroyed it is gone forever. If we disregard this, a time will come when there would not be enough soil left to sustain life on earth, because the soil is a necessary growth medium for plants, a home for certain insects and animals, as well as a medium from which we get minerals. It is therefore important to treat soil, especially topsoil, as a living entity.

Erosion is the process of detachment and transport of soil particles by erosive agents and it is caused when soil is removed through the action of wind and water. Erosion changes structure and texture of the soil. Therefore, soil erosion is a major agricultural and environmental problem, accelerated by deforestation, overgrazing and poor land management. Causes of soil erosion occur when farming practices are not compatible with the fact that soil can be washed away or blown away. These practices are overstocking and overgrazing, inappropriate farming techniques such as deep ploughing land 2 or 3 times a year to produce annual crops, lack of crop rotation, planting crops down the contour instead of along it, lack of proper land use system. Much of the eroded soil is deposited both in low areas of the field, or it moves off the farm and eventually enters drainage ditches, streams or river called *Chemoga*, which is a major tributary of Blue Nile River (Sutcliffe & Parks, 1999; Teferi et al., 2010).

Soils that enter this watercourse reduce water quality (Stuart, 1971), efficiency of drainage system and storage capacity of the river. All water surfaces are colorless but river Chemoga is colorful because during rainy season all soils from farmlands of Yebrage and its surroundings will be eroded and delivered to the river Chemoga. Due to this reason sometimes it becomes blue, sometimes reddish and sometimes dark brown so that the color of water changes seasonally. (Teferi et al., 2010) since the productive capacity of the cultivated land is being threatened by the loss of macro/micronutrient and soil organic matter through erosion/soil movement, farmers in Yebrage (study area) are looking for another options because they could not cope with the declining crop yields.

The loss of protective vegetation through overgrazing, ploughing and fire makes soil vulnerable to being swept away by wind and water. (Councile, 1994) and also increases the chance of leaching. Scarcity of vegetative cover of farmland will be the cause for the change of structure and texture of the soil. This is the reason for having most of the elements in very low amount.

Substances that rapidly degrade can be quickly removed from the environment. The absence of rapid degradation in the environment can mean that a substance in the soil has the potential to exert toxicity over a wide temporal and spatial scale.

The (Rose, 2001) experimental investigations were carried out at the scale of farmer practice at the chosen study sites by measuring soil nutrient content from hydrologically isolated farmland plot. From the conducted experiment it is concluded that the farmlands of Yebrage are deficient of soil fertility because they lack all the macro/micronutrient and organic matter which are very important for crop production and also plant growth.

Chapter 6

III.Thermal Neutron Activation Analysis of Rock Samples from Choke Mountain, East Gojjam, Ethiopia

6.1 Background:

Rock samples from Choke Mountain were subjected to elemental analysis by (NAA) technique with the aim of broadening any database information on their elemental composition and concentration and accessing the extent of their environmental friendliness. These observations suggest that all particulate emissions and wastes from the Choke Mountain range should be closely monitored to reduce their effects on the environment and health. This study demonstrates the principles of element identification using the technique of neutron activation analysis. The instrumental neutron activation analysis technique ($INAA$) (Sarathi et al., 2008) was used in the qualitative and quantitative analysis of rock samples from the Choke Mountain area in East Gojjam and it was an established analytical technique for determining trace elements in a wide variety of materials, including rocks and other geological samples. A significant advantage of (NAA) over other techniques is the simplicity of sample treatment before analysis: in most cases, the only requirement is that the sample would be reduced to a size suitable for encapsulation. With appropriate experimental parameters, excellent sensitivity is possible for some elements. The primary objective of this work is to determine and identify the various (Win, 2004; Abd et al., 2009) major, minor and trace elements present in rock samples of Choke Mountain. Their effect on the environment such as humanity, animals and atmosphere will be discussed.

6.2 Introduction:

The Choke Mountain is located to the south of Lake Tana in the Center of East Gojjam Administrative Zone. The nearest towns are Bahir Dar to the north and Debre Markos to the south. The Choke Mountain is the water tower of the region serving as headwaters of the upper Blue Nile basin. This study area is located in North−Western side of Ethiopia and covers the whole of the Choke Mountain range, the most important source of water for the Blue Nile river system in Ethiopia. Most of the tributaries of the Blue Nile River originate from this mountain range. Choke Mountain (CM) is a well defined massif that forms the northern limit of East Gojjam

Figure 6.1: One of the four peaks

Administrative Zone. CM is the coldest and most important ecosystem in East Gojjam. The total duration of the rainy season is about 5 months from June−October.

There is no longer significant natural forest cover in this mountain range. Since the productive capacity of the cultivated land is being threatened by the loss of nutrients through erosion, farmers in this area are looking for another option because they could not cope with declining crop yields. Every year more and more agricultural land is being converted into Eucalyptus forest plantations which ensure income security for residents.

This tends in land use and land cover change is altering the soil and hydrologic characteristics of upland watershed of the Choke Mountain range. This may also influence the livelihood of the population living in downstream areas of changing critical watershed functions. The primary objective of this work is to determine and identify the various major, minor and trace elements (Akpan et al., 2010) present in rock samples of Choke Mountain, and also to qualify nutrient dynamics and estimate nutrient loss in the choke mountain range. The rocks of this area are mainly sedimentary and metamorphic.

6.3 Materials and methods:

6.3.1 Sample collection and preparation:

Three representative samples were collected from Choke Mountain range near Debre Markos in East Gojjam Administrative Zone, Ethiopia, between October, 2010 and February, 2011. ($C1$) for the samples from the northern side of the Choke Mountain range, ($C2$) for the samples from the southern side of the Choke Mountain range and ($C3$) for the samples from the eastern side of the range were assigned to the samples for proper identification.

Searching for information on this area, I found that there has been quite a bit of research on “habitat loss” such as recent article in Hydrology and Earth Science journal with the intriguing title “the use of remote sensing to quantify wetland loss in the Choke mountain Range, Upper Blue Nile Basin, Ethiopia” (Teferi et al., 2010).

The source of the Blue Nile River and its tributaries, and this area had quite a few

Figure 6.2: The source of Blue Nile River

springs, being surprised with their subtle beauty. Most emerged from nowhere, creating little muddy streams with flattened yellow flowers. But there were sheep grazing at this high altitude, so most of the muddy streams became trampled with little sheep hooves and started to resemble wallowing areas. It was also more impressed with the rocks, lichens, and moss than everything else.

Figure 6.3: The area at which C1 is collected

There are really cool endemic trees here. Based on this incident, it is pretty obvious that it would be much better for what is left of this unique environment if nobody comes out here at all.

There are highly attractive and charming short day plants, which behave both annual as well as perennial flowering herbs. The leaves are alternate and toothed, roots are adventitious and the stems are woody solids. The flowers bloom in early winter with a wide range of colors, shapes and sizes. The flowers colorrange from white and cream through the shade of yellow, pink, bronze, red, deep purple and green.

Figure 6.4: The area at which C2 is collected

The most remarkable feature of this mountain is the virtual absence of forest. The major natural habitats are moist moorland with giant Lobelia spp., sedges and tussocks, Festuca spp., and other grasses, montane grass lands and meadows, cliffs, and rocky areas.

Figure 6.5: The area at which C3 is collected

The mountains are totally unprotected and are severely threatened by the rapid agricultural expansion, overgrazing and excessive soil erosion. The “almost threatened” Abyssinian Longclaw lives in this area. It resembles a meadowlark in many ways.

The exact location of this research site in terms of latitude, longitude and elevation above sea level are: $(10^{0}34'40"N)$ latitudes, $(37^{0}50'31"E)$ longitudes and their elevation varies from (3300-3950) meter above mean sea level ($amsl$). Its annual mean temperature is below 10 °C. The location and geological map of the Choke Mountain as well as the sampling map of the investigated samples are shown in the $fig.(6.3, 6.4, 6.5)$.

The sample locations that were determined by the use of GPS were plotted on the geological map of the study area (see Maliszewska−Kordybach et al., 2008; Angelal et al., 2010; Kijowska, 2004, chap. 3).

The Choke Mountain rock samples were collected at distances of about 200 m from each other since the range is very extensive.

6.4 Experimental measurements performed at NAA:

The fitted spectrum and the experimentally derived continuous background shown in $fig.$(6.6) are typical of an NAA spectrum. According to (Obiajunwa & Nwachukwu, 2000; Mokobia et al., 2006), the observed continuous background was an inherent property of all NAA spectrum. The strengths of the detector signal which are proportional to gamma−ray energies were processed and converted to digital form for storage on a multi−channel analyzer at the end of the measurements.

The accuracy of (NAA) technique had been checked by carrying out replicate analysis of standard materials having certified values. Good agreement between experimental values and previously published working values was observed for most of the elements and reference materials analyzed during this study when analysis was done before and after irradiation.

Background Optimization: $Fig.$(6.6) is the detection room background spectrum while Fig.(6.7, 6.8 and 6.9) show the net spectrum of the activated samples by subtracting the room background. Energies of various gamma−rays observed and also marked at different peaks (see chapter four).

$*K^{40}$ and $*Pb^{212}$ were background radiations and they were available inside the samples and also as natural radioactivities.

The masses and concentrations of these gamma−rays would not be not calculated because of their long half−lives. (Some gamma peaks are not shown on the figures above since they are not seen properly.)

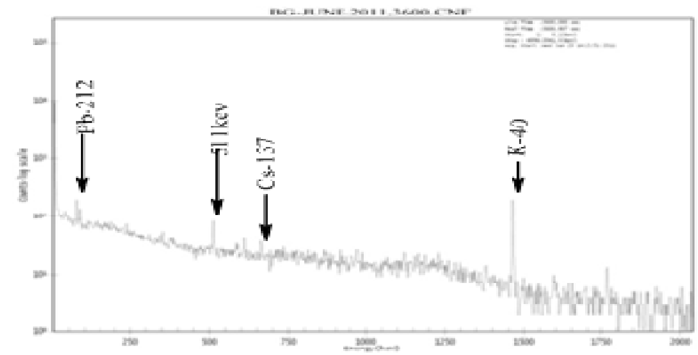

Figure 6.6: Background spectra of the counting room

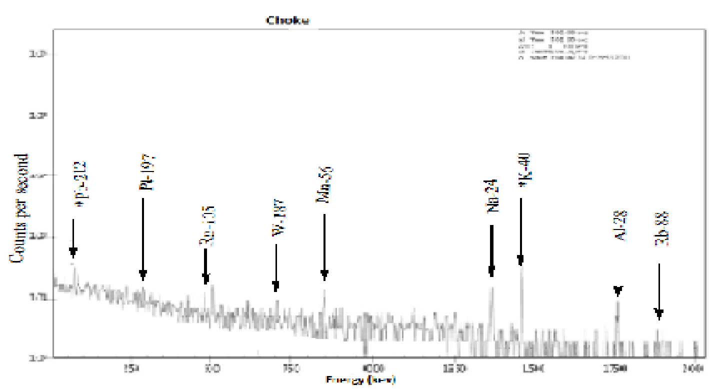

Figure 6.7: Gamma spectra of rock sample for Choke Mountain (C1)

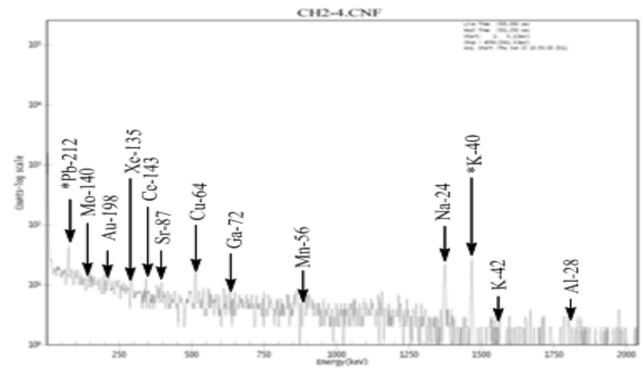

Figure 6.8: Gamma spectra of rock sample for Choke Mountain (C2)

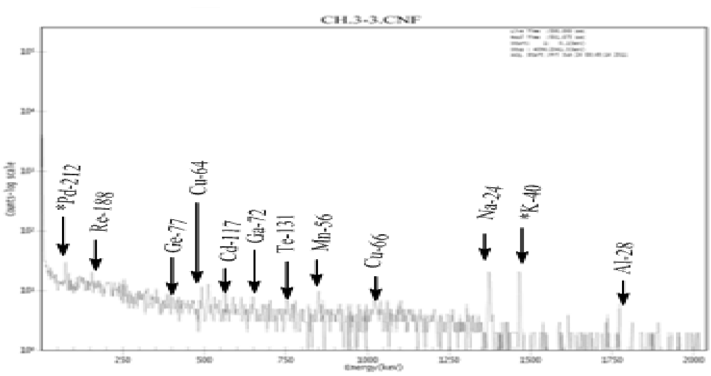

Figure 6.9: Gamma spectra of rock sample for Choke Mountain (C3)

Decay Scheme:

Some decay schemes of the radioisotopes are:

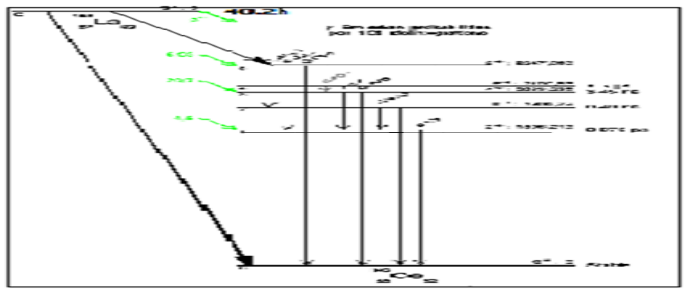

Figure 6.10: Decay Scheme of La-140

La^{140} decays by beta minus emission to the Ce^{140} excited levels.

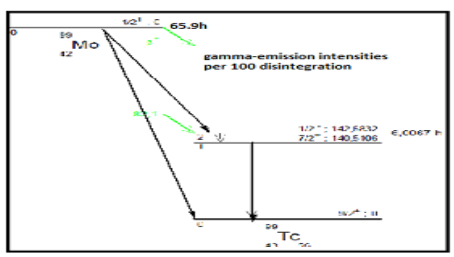

Figure 6.11: Decay Scheme of K-42

K^{42} disintegrates to the Ca^{42} excited levels by beta minus emissions.

Half-life and decay constant determination:

Sample decay curves:

Using the same expressions of equations (4.2.1) and (4.2.3), the decay constants and half−lives of the curves could be calculated (sea chapter four).

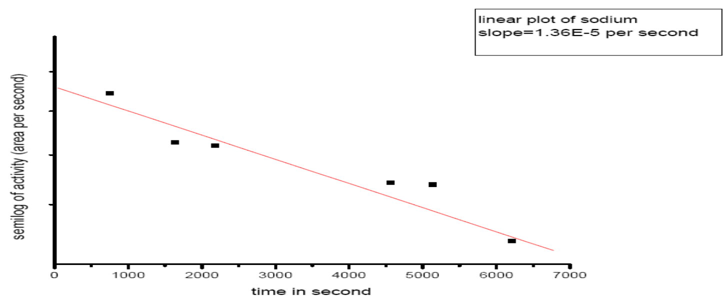

Figure 6.12: The exponential decay of activity of sodium in semilog plot

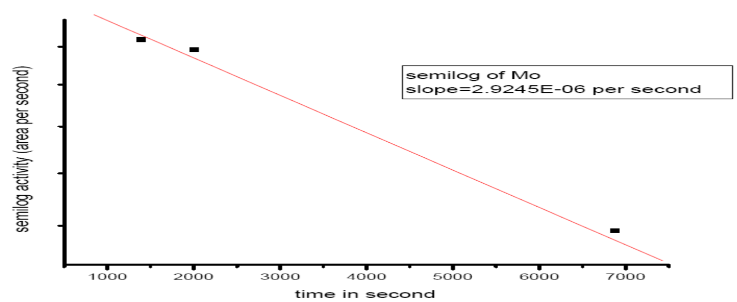

Figure 6.13: The exponential decay of activity of Mo in semilog plot

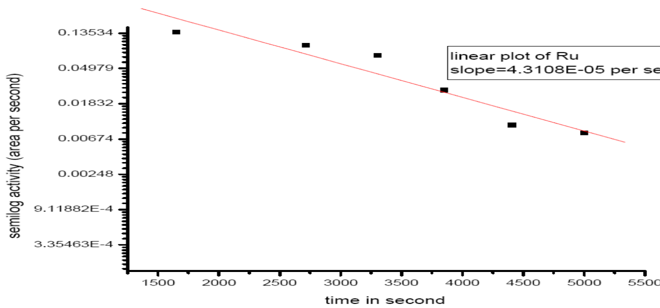

Figure 6.14: The exponential decay of activity of Ru in semilog plot

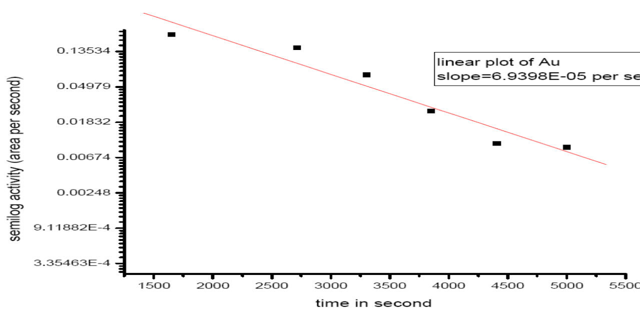

Figure 6.15: The exponential decay of activity of Au in semilog plot

Experimental identification and quantification:

Element concentrations in rock samples by using (NAA) are shown in the table below. This table summarizes the concentration of detected elements of the Choke Mountain range rocks.

Table 6.1: Elements obtained from rock samples collected from the Choke Mountain range using Neutron Activation Analysis (Irradiated for about many days with known standards: Potassium Iodide)

Radio Nuclide	I_γ (%)	half life	f_γ (%)	E_γ (kev)	Concentration(ppm)		
					C1	**C2**	**C3**
Al^{28}	100	2.24m	100	1778.9	41.25±0.25	1.08±0.047	0.8426±0.049
Na^{24}	100	14.96h	100	1368.6	86.47±0.42	197.08±0.63	145.123±0.54
Mn^{56}	100	2.56h	99	846.7	0.052±0.03	0.474±0.031	0.0418±0.009
Rb^{88}	27.83	17.7h	21	1836	76.85±0.39	ND	ND
La^{140}	99.9	1.678d	95	1596.2	0.049±0.0095	ND	ND
Cu^{64}	69.1	12.7h	37	511	0.353±0.027	2.69±0.07	ND
Cu^{66}	30.9	5.25	8	1039.2	1.431±0.053	ND	2.17±0.03
Eu^{152}	47.77	9.31h	43	970.35	0.034±0.0082	ND	ND
Cr^{51}	4.35	3.5m	10	320.08	6.39±0.11	ND	ND
Ge^{77}	36.4	82.78m	1	465.6	26.13±0.23	ND	ND
Ru^{105}	18.6	4.44h	17	469.37	70.97±0.38	ND	ND
W^{187}	28.4	23.72h	25	685.77	0.47±0.03	ND	ND
As^{76}	100	1.0778d	6	657.04	8.913±0.13	ND	ND
Cd^{117}	7.6	3.31h	26	1997	58.53±0.34	ND	0.025±0.010
K^{42}	6.7	12.36h	18	1522.9	ND	18.01±0.19	ND
Mo^{99}	24.4	66h	5	140.2	ND	378.02±0.9	ND
Re^{188}	62.93	16.7h	15	155	ND	5.501±0.011	196.2±10
Au^{198}	100	2.7h	95	412	ND	0.00014±0.0001	ND
Sr^{87}	9.9	2.8h	83	388	ND	0.26±0.023	0.84±0.06
Ga^{72}	40	14.1h	25	626.2	ND	4.325±0.009	4.71±0.23
Te^{131}	34.5	3.0h	50	779.2	ND	ND	0.02±0.01
Ge^{77}	7.7	11.3h	21	419	ND	ND	0.06±0.01
Xe^{135}	10.4	9.14h	90	248	ND	1.9±0.0004	ND

$*ND$ = not detected.

The knowledge of specific activities or concentrations and distributions of radionuclide in these materials are of interest since they provide useful information in the monitoring of environmental radioactivity.

6.5 Results and Discussions:

A non−destructive, extremely sensitive, accurate, quick and cost effective Neutron Activation Analysis technique (James & Cynthia, 2002; Berlizov, 2009) was applied with success to determine the concentration values of the most elements in such complex rock samples (Soliman, 2006). It is generally one of the most sensitive and non−destructive techniques used in the identification of elemental concentrations.

The elemental composition of Choke Mountain Rock has been determined using this NAA technique. Altogether thirteen elements (Al, Na, Cd, Rb, Ge, Ru, Cu, W, Cr, As, Eu, La, and Mn) from ($C1$), eleven elements (Al, K, Mo, Re, Au, Sr, Ga, Na, Xe, Cu, and Mn) from ($C2$) and ten elements (Al, Cd, Re, Sr, Ga, Te, Na, Mn, Cu, Ge) from ($C3$) were observed in the choke mountain range. K was observed in ($C2$) rock sample. Some harmful rare earth elements (Rb, Mn, As, Sr, Cr, Cd, Te, Mo, Cu,) (Ljung et al., 2011) were observed in the rock samples. These elements are unfortunately notorious for being common contaminants as they are very abundant elements.

6.5.1 Conclusion:

Speciation (James & Cynthia, 2002) analysis is not performed on a variety of sample types using High Performance Germanium detector to separate different forms of an element. Thus all wastes and particulate emissions from the Choke Mountain range should be closely monitored to reduce their adverse effects on health and environment (Aigbedion & Iyayi, 2007).

Accuracy and quality assurance was controlled by the results obtained from the analysis of standard samples. These results suggest that care must be exercised in comparing the data obtained from the whole rock samples of Choke Mountain with various NAA procedures.

Chapter 7

V.Instrumental Neutron Activation Analysis of Rock Samples from Blue Nile Gorge, East Gojjam, Ethiopia

7.1 Background:

Neutron Activation Analysis (NAA) is an analytical technique based on the measurement of characteristic radiation from radionuclides formed directly or indirectly by neutron irradiation of the material of interest. The major objective of this work is to determine and identify the various major, minor and trace elements present in the rock samples from Blue Nile Gorge.

The hydrocarbon potential of the Paleozoic sediments is much debated in this Grand Canyon and the hydrocarbons were frequently re−mobilized and redistributed into younger sediments. Paleozoic rocks could have produced significant amount of hydrocarbons in areas characterized by strong Mesozoic sedimentary subsidence.

The recent developments in activation analysis techniques now offer the prospect of rapid, reliable analysis for many elements in concentrations as small as parts per million or parts per billion. The neutron obtained from isotopic neutron source of (α, n) type was used in this research paper.

7.2 Introduction:

Ethiopia is in the tropical zone laying between the Equator and the Tropic of Cancer, located in the horn of Africa and a largely (Sileet, 2001) mountainous country, which is the origin or source of much of the rivers flow reaching the Blue Nile River (Awulachew et al., 2008; Sutcliffe & Parks, 1999; Steenhuis et al., 2009) contributing greater Nile flow (Melesse et al., 2004; Gebremariam, 2010) possibly increasing during the rainy season (Amy et al., 2004). The central high−lands with altitudes between $1,500\ m$ and $4,000\ m$ are distributed by numerous rivers, including the Blue Nile River (Gebremariam, 2010; Sutcliffe & Parks, 1999).

The economy of Ethiopia which is primarily based on Agricultural production and farming is a major economic activity of the people (Quarshie et al., 2011) in the area where the main food crops are “teff” (Eragrostis Teff), which is a small grain, cereal crop, wheat, barely, sorghum, millet, yams, potatoes, and beans. The mineral potential of Ethiopia lies mainly with the development of gold, potash, thermal energies and industrial materials.

Ethiopia has a varied geology and spectacular topography with the major East Africa rift cutting its plateau (Beaulieu & Gaonaćh, 2002; Gebremariam, 2010). The Blue Nile Gorge (the River Abay Gorge as locally known) containing a complete stratigraphy of the Ethiopian geology starting with a Precambrian rock unit at the base overlain by a thick sequence of Mesozoic sediments (Gani et al., 2008; Sileet, 2001; Baba, 2000; Hagos et al., 2010) capped by the tertiary trap basalt making up the Ethiopian plateau. The thickness ranges from about 100 m to 700 m. In their area they are about 300 m thick and outcrops on both sides of the river, forming nearly vertical cliffs. For a million of years the Blue Nile has been carving this huge gash through the Ethiopian high lands. The Blue Nile Gorge, also known as Africa's Grand Canyon (Ayalew & Yamagishi, 2003), one of the nicest canyons in Africa and located in Ethiopia, is over $1,000$ m deep, nearly 20 km wide and over 600 km long.

From all over the highlands of Ethiopia including Choke Mountain (Teferi et al., 2010), huge rivers pour into the Blue Nile Gorge. By the time it leaves Ethiopia the Blue Nile River will be 50 times the size (Amy et al., 2004) it was in the dry season. A large portion of the (Königshof & Boncheva, 2005) Paleozoic and Mesozoic sediments (Baba, 2000; Hagos et al., 2010) in the Blue Nile basin are buried under thick Tertiary Volcanic. Along the western margin of the north$-$western plateau in the Blue Nile Gorge, a 2000 m section of Mesozoic strata (Königshof & Boncheva, 2005)

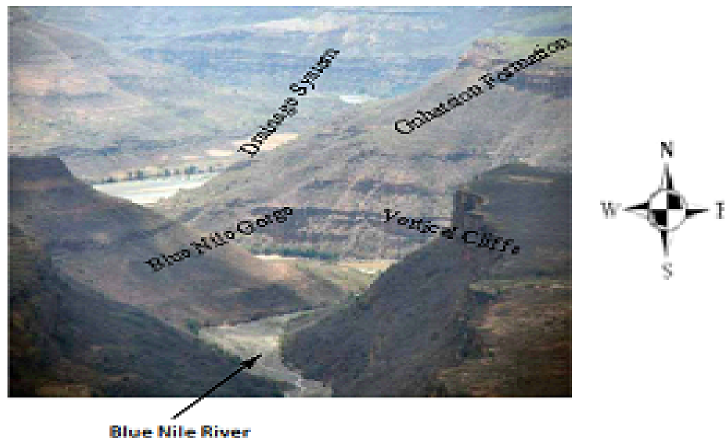

Figure 7.1: Satellite map of the Blue Nile Gorge/Africa'Grand Canyon showing topography and drainage pattern

capped by massive tertiary volcanic is exposed. The basement of Ethiopia consists of metamorphic and igneous rocks of Precambrian and lower Paleozoic age. Between the Ordovician and Early Mesozoic system of northeasterly as well as northwesterly trending troughs were filled with continental sediments (Mogessie et al., 2002).

Mesozoic sediments (Ayalew & Yamagishi, 2003; Baba, 2000; Hagos et al., 2010) are widely deposited in Ethiopia during a continuous subsiding period of the land and migration of the sea from East, covering the central part and northern areas of the country, and are exposed on the central dissected plateau areas (Steenhuis et al., 2009) in the Blue Nile River basin. The Mesozoic sediments (Baba, 2000) are important for their associated industrial and building materials including limestone, sand, sandstone, gypsum and clay. Total reserves are enormous because the thickness of the gypsum deposits in many hundreds of meters and the formations are known to extend laterally for hundreds of kilometers.

The Paleozoic and Mesozoic sediments (Hagos et al., 2010) are the bases for discordant layers of thick massive flood lavas, mainly basalts, which are generally post−Oligocene in age and reach a maximum thickness of 5500 m. The typical Mesozoic succession of the basin is about 1200 m thick and includes from bottom to top many formations. Gohatsion Formation is represented by greenish, grey or brown colored dolostones of about $(50-80)cm$ thick and shales. The dolostones are characterized by flute casts at the base of beds, ripples and flasher bedding at the lower part and parallel lamination at the top. At the top occur mud−stones with thin layers of angular quartz including fossils like bivalves and gastropods, further fine siltstone with small bivalves and gastropods, fine−grained sandstones with cross−lamination at the base and parallel lamination at the top. The upper part is characterized by fossil free, green, red and brown clays and siltstone covered by dolostones.

The Blue Nile Gorge is situated 225 km north of Addis Ababa. This majestic and enormous gorge is the most captivating gorge in Africa. It has a magnificent, captivating physical feature, natural beauty and a unique eco−system. The Blue Nile Gorge is a tremendous obstacle for traveling and communication from the northern half to the southern half of Ethiopia.

The river (Teferi et al., 2010; Awulachew et al., 2008) got the "blue" part of its name because of its muddy color due to the huge amount of fertile soil it erodes (Yihunie et al., 2011) from the Ethiopian highlands. Why they called it blue rather than the more logical color of brown, dark brown or even black is open to conjecture. It is this spectacular flood of the Blue Nile that sets it apart from many great river systems of the world. Each year during the heavy rains, the (Steenhuis et al., 2009) Blue Nile swells to over 50 times its dry season size and carries with it a staggering 140 million tones of rich, fertile silt (Zhao et al., 2011) as its rages, thick and brown, towards the Mediterranean Sea.

The Abay river section is exposed along a road cut at the Addis Ababa to Debre Markos highway, between kilometer marks 198 and 215. This road connects Addis Ababa to the northern part of the country which passes through the gorge of Blue Nile where there is a high susceptibility land−sliding. Rock falls on the other hand exist largely as discernible block topples and wedge failures all along mountains, valley walls and road cuts (Ayalew & Yamagishi, 2003).

The main objective of this study is to assess elemental availability and concentration in rock samples of the (Wolela, 2008) Blue Nile Gorge area between Gohatsion and Dejen towns. Due to population growth, demand and shortage of land resources (Awulachew et al., 2008), farmers tend to use sloppy terrains, for settlements, cropland and infrastructure construction along this gorge (Vitale & Lee, 2005) using poor land management practices since agricultural investments are difficult to make in this ecosystem due to natural barriers. Gohatsion and Dejen are two small towns located just outside the gorge in the South and in the North sides of the gorge respectively. The study area can be reached easily using the asphalt road that goes from Addis Ababa to the northern part of the country. The road passes through the gorge crossing the two small towns in the north and south edge of the valleys.

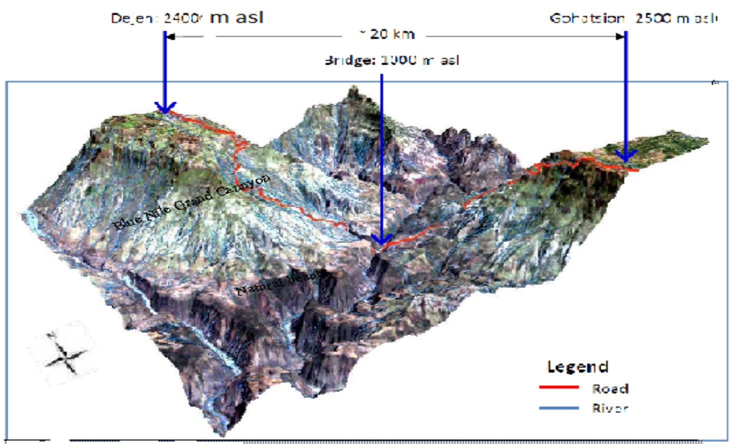

Figure 7.2: View of the study area showing topography and drainage pattern

Sometimes landslide destructs retaining walls and breaks the road hindering and interrupting traffic movements.

The Blue Nile River and its tributaries cut deep into the rocks (Ayalew & Yamagishi, 2003) forming spectacular valleys with vertical cliffs and deep gorges in which landslide problem is common in the area mainly during the rainy season. The Blue Nile passes through deep valley and gorge as a raging torrent during and after the wet seasons conveying large sediment loads (Sileet, 2001).

7.2.1 Research question:

What is the effectiveness of NAA to identify elemental content of selected geological samples from specific areas of East Gojjam regardless of oxidation state, chemical form or physical location ? Using this central research question the following sub−question is developed. Which samples are collected, how they are collected and what are the analytical techniques that are applied to them for the entire study ?

7.2.2 Sample Collection and Preparation:

A total of 3 rock samples have been collected randomly from different sites in Blue Nile Gorge. The samples were stored (Choudhury et al., 2006) in pre−cleaned polyethylene capped bottles and handled with extreme care until the moisture is completely removed from the samples' surface. The sample locations were determined (see

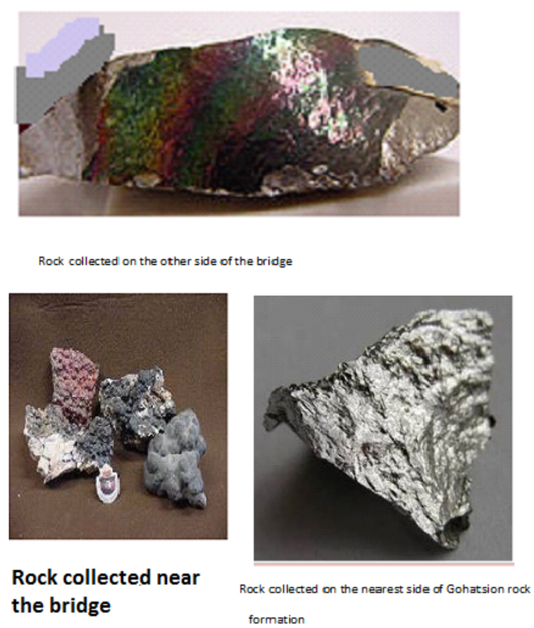

Figure 7.3: Rock samples collected from Blue Nile Gorge

Maliszewska−Kordybach et al., 2008; Angelal et al., 2010; Kijowska, 2004, chap. 3).

7.3 Experimental Measurements performed at NAA:

7.3.1 Background Optimization:

(see chapter four).

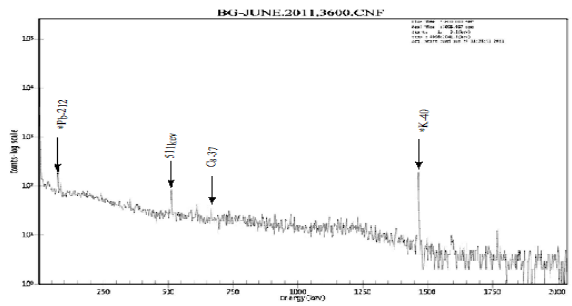

Figure 7.4: Analysis of Gamma Spectra for Room Background

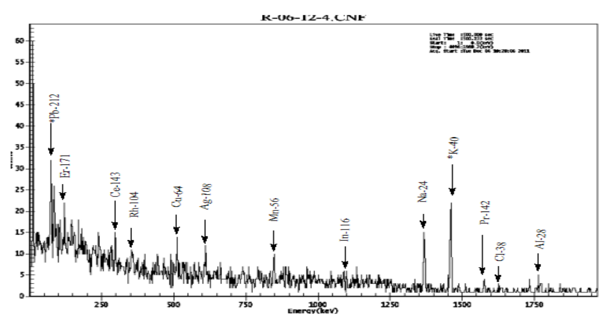

Figure 7.5: Analysis of gamma spectra for rock samples from Blue Nile Gorge obtained by irradiation with isotopic neutron source

Since ($*K^{40}$ and $*Pb^{212}$) are found inside the building materials of the room, their peaks never decay as such, and other photopeaks are not shown on the spectrum too.

The photopeaks in the spectrum indicate the presence of particular radioelement, and the amount of radioactivity they contain may be used to measure the amount of the radioelement. Each spectrum was collected in the live−time. A qualitative analysis may then be performed by identifying the peak energy values as well as a qualitative analysis, by determining the peak area proportional to the radioisotope activity (absolute measure).

Decay scheme: Decay schemes of some of the gammas: Mg^{27} decays 100% by

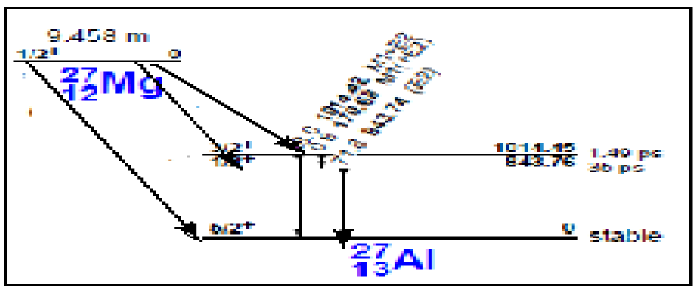

Figure 7.6: Decay scheme of Mg

beta minus emission to Al^{27}. A significant fraction directly populates to the ground state.

The Mg^{27} decay curve gave a half−life of 9.45 minute and a graphical extrapolation to the end of the irradiation was made to obtain the counting rate at zero time.

For Au, the following energies were found in (kev): (411.8, 675.9, 1087.7) which it was attributed as belonging to Au^{198} and for which the disintegration scheme is illustrated in fig.(7.5). The transitions measured in this work are denoted by heavy lines and the energies of the levels are obtained from present measurements. The Au^{198} disintegrates following three beta minus transitions, the main one is to the 411 kev level of Hg^{198}. The vertical axis depicts the energy of the parent as being the

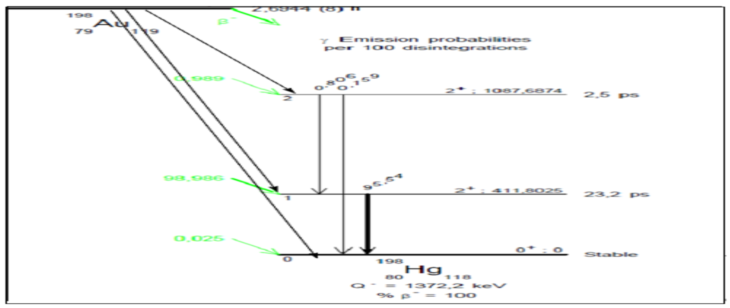

Figure 7.7: Decay scheme of Au-198

highest and with the resulting decay: the energy decreases until the daughter nucleus reached 0 Mev making it the ground state.

Decay scheme of Cl:

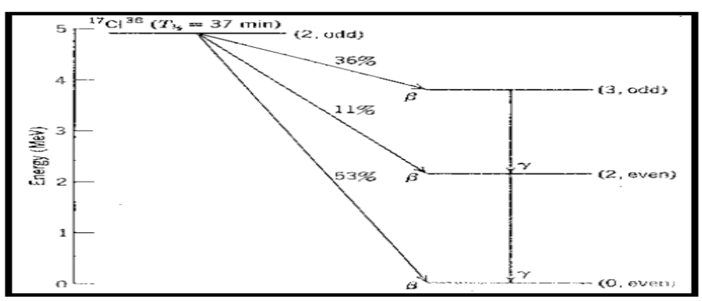

Figure 7.8: Decay scheme of Cl-38

Experimental elemental identification and half-life determination:

Sample decay curves:

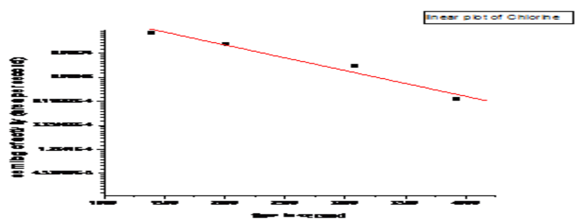

Figure 7.9: The exponential decay of activity of Cl in semilog plot

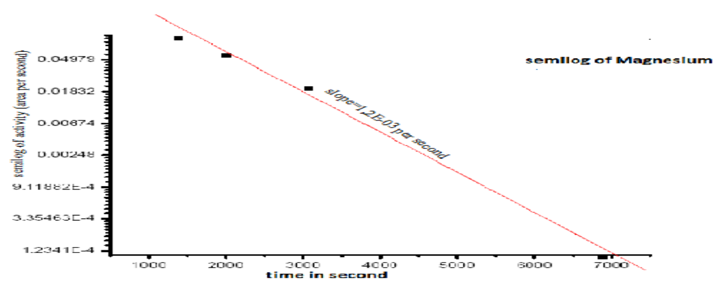

Figure 7.10: The exponential decay of activity of Mg in semilog plot

The shape of curves like these are exponential and so radioactivity exhibits exponential decay.

Using the same expressions of equations (4.2.1) and (4.2.3), the decay constants and half−lives of the curves can be calculated (sea chapter four).

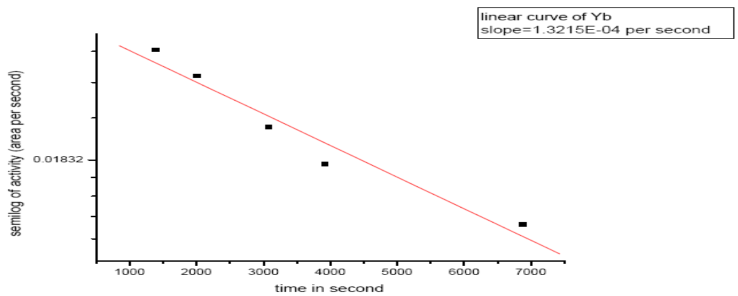

Figure 7.11: The exponential decay of activity of Yb in semilog plot

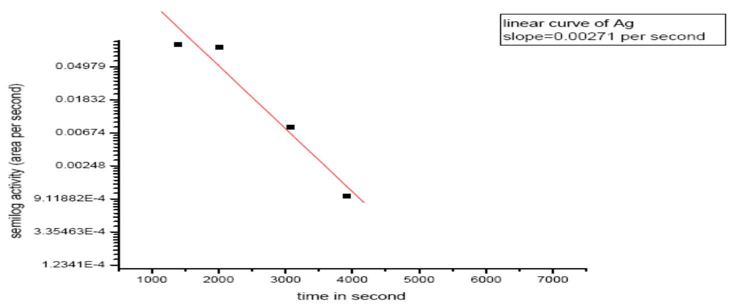

Figure 7.12: The exponential decay of activity of Ag in semilog plot

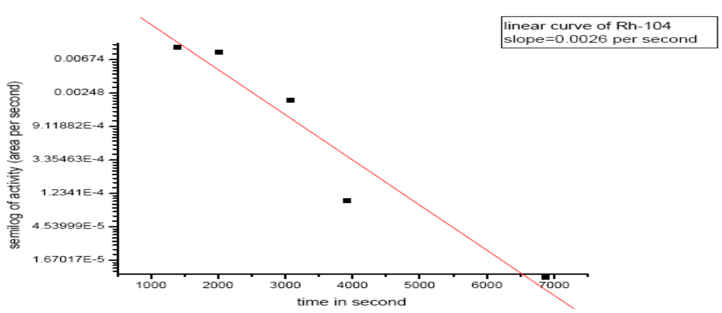

Figure 7.13: The exponential decay of activity of Rh in semilog plot

7.3.2 Quantitative analysis of NAA:

Elemental identification and concentration:

Table 7.1: Analysis of Radionuclide of the Rock Sample from Blue Nile Gorge

The radio nuclide	Natural Abundance in(%)	The Half life	Concentration in (ppm)	Gamma energy in (kev)	Gamma abundance in (%)
Er^{171}	14.88	7.52h	2.347±0.009	124	9
W^{187}	28.4	23.9h	3.623±0.09	134	9
Yb^{177}	12.73	1.9h	4.36±0.093	150	20
Ce^{143}	11.07	33h	118.72±0.49	293	42
Ru^{105}	18.6	4.44h	60.059±0.35	469	17
Ag^{108}	51.83	2.41m	0.4447±0.03	619/434	9/17
Mg^{27}	11.01	9.46m	86.369±0.42	844	72
Mn^{56}	100	2.56h	6.4458±0.11	847	99
Rh^{104}	100	4.41m	0.23099±0.021	357	13
In^{116}	95.7	54m	0.01896±0.006	1097	56
Na^{24}	100	15.03h	2.7048±0.07	1369	100
Pr^{142}	100	19.2h	3.1138±0.08	1576	4
Cl^{38}	24.23	37.18m	14.289±0.13	1642	32
Al^{28}	100	2.246m	135.502±0.52	1779	100
Cu^{64}	69.1	12.7h	93.673±0.43	511	37

Using bar graph, the concentrations of the radionuclides are analyzed as they are depicted in fig.(7.14).

From table (7.2), major elements include (Cu, Al, Cl, Ce, Mg, and Ru), minor elements include (W, Er, Yb, Mn, Na, Pr ,Rh, and Ag) and trace element includes (In).

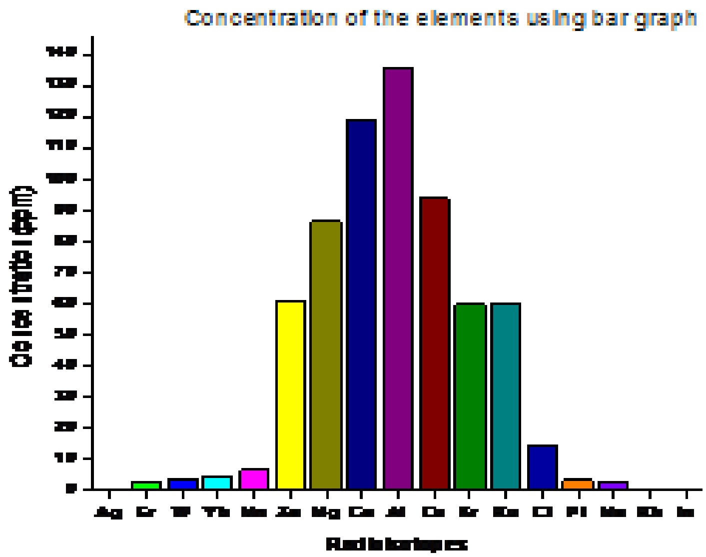

Figure 7.14: Concentration of the radionuclides using bar graph

Table 7.2: Concentration of major, minor and trace)

Stable isotope	I_γ (%)	Concentration(%)		
		Major	**Minor**	**Trace**
Cu^{63}	69.1	9.37		
Al^{27}	100	13.55		
Cl^{37}	24.23	1.43		
Ce^{142}	11.07	11.87		
Mg^{26}	11.01	8.63		
Ru^{104}	18.6	6.02		
W^{186}	28.4		0.364	
Er^{170}	14.88		0.234	
Yb^{176}	12.73		0.636	
Mn^{55}	100		0.645	
Na^{23}	100		0.271	
Pr^{141}	100		0.311	
Rh^{103}	100		0.023	
Ag^{107}	51.83		0.0445	
In^{115}	95.7			0.0006

7.3.3 Errors in the measurements:

The statistical error of counting, mass measurement, and all other different measurements were estimated to be around 10%. The possibility of other nuclide gamma−ray emission contribution to the analyzed peak was considered. The electronic dead time in all measurements was less than $(2 - 5)\%$.

7.4 Results and Discussions:

A non−destructive neutron activation analysis (Kogo et al., 2009; Srinivas, 1995) technique was applied with success to determine the concentration values (Arakawa et al., 2003) of the most elements in such complex samples (Soliman, 2006). Some gamma−ray lines appeared in the spectra, due to background emitted from the shielding materials. These have been subtracted from each spectrum by using an empty container and measured at the conditions of samples measurement.

Sensitivities and accuracy of (NAA) depend on the concentration of the element and radionuclide parameters (i.e, parent isotope abundance, neutron cross−section, half−life, and gamma−ray abundance).

It was noticeable that by using (Daraban et al., 2005) the isotopic neutron source (Tohamy et al., 2010), some radioisotopes with great importance in Environmental Science could be produced: (Cu, Al, Cl, Pr, Na, In, Rh, Mn, Mg, Ag, Ru, Ce, Yb, W, Er) including the background elements have been appeared in the measured spectra. The elements (Cu^{63}, Al^{27}, Cl^{37}, Ce^{142}, Mg^{26}, and Ru^{105}) are found as major elements, the elements (W^{186}, Er^{170}, Yb^{176}, Mn^{55}, Na^{23}, Pr^{141},Rh^{103} and Ag^{105}) are found as minor elements and the element (In^{115}) is found as trace element.

The $INAA$ technique (Sarathi et al., 2008) has lead to a successful determination of fifteen elements in the rock samples around the research area. Also, many radioisotopes produced in this way were studied, with different applications (Daraban et al., 2005) as heavy metals like (Al, Er, Mn, Cu, Mg, Ag, Pr) among others. Some of these heavy metals have been known to be (Zukowska & Biziuk, 2008; Leszczynska & Ahmad, 2006) toxic and dangerous to human health especially when they are found in ground water and growing crops.

Some values of their concentration (Arakawa et al., 2003) were as high as (2.347 ± 0.069 ppm for Er^{171}, 3.623 ± 0.09 ppm forW^{187}, 4.363 ± 0.093 ppm for Yb^{177}, 118.72 ± 0.49 ppm for Ce^{143}, 60.059 ± 0.35 ppm for Ru^{105}, 0.447 ± 0.03 ppm for Ag^{108}, 86.369 ± 0.42 ppm for Mg^{27}, 6.4458 ± 0.11 ppm for Mn^{56}, 0.23099 ± 0.021 ppm for Rh^{104}, 0.01896 ± 0.006 ppm for In^{116}, 2.7048 ± 0.07 ppm for Na^{24}, 3.1138 ± 0.008 ppm for Pr^{142}, 14.289 ± 0.17 ppm for Cl^{38}, 135.502 ± 0.52 ppm for Al^{28} and 93.6735 ± 0.43 ppm for Cu^{64}) and the elements (Ce, Pr, Er, and Yb) are rare earth elements (El−Tahar, 2006) found inside the rock sample. Everyday we are exposed to hundreds of toxic metals and chemicals.

The heavy metals that most commonly cause problems include (Al, Cu, and Mn). All heavy metal poisoning and chemical toxicity lead to the accumulation of toxins in human tissues and organs causing nutritional deficiencies, hormonal imbalances and the breakdown of the immune system, the central nervous system, and the organs of the body. Some of these elements accumulate in the body, so there is no truly safe exposure limit for these elements.

As with the other elements, aluminum is observed and accumulated in the body, and has been linked to serious illnesses including osteoporosis, extreme nervousness, anemia, headache, decreased liver and kidney functions, forgetfulness, speech disturbances and memory loss (Morcola & Droese, 2003).

The man−made elements are radioactive and toxic whether they are metals or not. Everything is toxic if the dose is high enough. A huge number of gamma−ray lines could be identified in each spectrum for each sample.

In the experimental investigation, Magnesium was found to be an essential element for plant growth and plays a key role in photosynthesis and other critical pathways in the plant. Chlorophyll, the green coloring in plants, is a magnesium−containing compound. Sufficient and balanced magnesium in the soil is needed to provide the optimum turf−grass performance and weed suppression.

A special computer program is used for identifying the elemental constituents of each sample under investigation. Data were manipulated (Dias & Prudêncio, 2007) using a variety of methods including spreadsheet/or excel. Due to its (Trkov, 2007; Sansoni, 1987) selectivity and sensitivity, (NAA) occupies an important place among the various analytical methods.

Chapter 8

Conclusions and Recommendations

8.1 Background:

This Chapter aims to draw general conclusions from the research work, critically evaluate findings, and make recommendations for future research. It provides a bird's−eye of key results in relation to the research questions and the overall objectives.

The study presented a multi−element analytical technique based on nuclear properties, Instrumental Neutron Activation Analysis ($INAA$), and in particular, the wide range of intensity applications that are possible with this technique. The different applications presented in this paper highlighted the main characteristics of the Neutron Activation Analysis, namely as a primarily analytical method and multi−elemental analytical techniques, it allowed the simultaneous qualitative and quantitative analysis of around fifty elements on an environmental sample, the manipulation and/slash or contamination of the sample under study. Even though neutron activation offered a great potential and a wide range of possible applications, it must be also noted that sometimes this technique needs to be associated to other analytical techniques that determine elements that may not be analyzed by activation and/or are not easily analyzed because of a reduced sensitivity.

8.2 Introduction:

This research work began with the broad central research question: "What is the effectiveness of the NAA to identify elemental content of selected geological samples from specific areas of East $Gojjam$ regardless of oxidation state, chemical form or physical location?" From this guiding question, the aim was to contribute to the body of knowledge of environmental science research and produce theory that had applicability to the development domain of the country.

Chapter one, Chapter two, Chapter three, and Chapter four, Chapter five, Chapter six and Chapter seven, provided a blueprint for the study undertaking.

8.3 Discussion of Results:

With the experimental framework in place the (Dierick, 2005) actual experiments can be considered. The main goal was to become familiar with the technique and its possibilities to characterize the setup and to explore new possible applications.

In Chapter four, results show that (NAA) of alluvial soil samples around the study area gave values of (Mn^{56}, Na^{24}, Al^{28}, W^{187}, Cu^{64}, As^{76}, Ru^{105}, Cd^{117}, Yb^{177}, Ir^{194}, Eu^{152}, Ba^{139}). Among the elements heavy metals include (Yb^{177}, Mn^{56}, Cu^{64}, Ru^{105},

Na^{24}, and As^{76}). Some of these metals have been known to be toxic and dangerous to human health specially when found in water and growing crops.

Arsenic, which is a semi−metallic element, is known to be strongly poisonous, especially when it is present in edible crops, through their roots.

Manganese, which is stored mainly in the liver and kidneys, poisoning has been linked to impaired motor skills and cognitive disorders. In the human brain the manganese is found as manganese metalloproteinase most notably glutamine syntheses in astrocytes.

A non−destructive neutron activation analysis technique (Dias & Prudêncio, 2007) was applied with success to determine the concentration values of the most elements in such complex samples.

In Chapter five, results show that (NAA) of soil samples around the study area gave values of (Er^{171}, Mn^{56}, Pd^{111}, Cu^{64}, Ga^{72}, Ta^{182}, Zn^{69}, Au^{198}, Xe^{135}, Sc^{46}, Cd^{115}, Mo^{99}, Al^{28}, La^{140} and Na^{24}).

It is explained why it is important, and introduce some important concepts and elements in the soil. The practical purpose of the soil is to enable more numerous, accurate and useful predictions (of land performance) to be made for specific purposes than could have been made otherwise (in the absence of location−specific information about soils). The study of soil for its own sake, as a natural object of interest, is worthwhile and extremely interesting, and if society as a whole or enthusiasts will support it. The primary objective of this section was to identify and quantify the various major, minor and trace elements present in the agricultural soil.

Environmental health hazards contains an analysis of morbidity and mortality data, continuity negative health factors (morbidity and mortality factors), their visualization, together with seeking relationships between the state of the natural environment and human health.

Heavy metals among the elements include (Er^{171}, Mn^{56}, Cu^{64}, Pd^{111}, Ga^{72}, Ta^{182}, Zn^{69}, Au^{198}, Sc^{46}, Cd^{115}, La^{140} and Mo^{99}). Some of them may be useful or toxic.

Palladium: is used in the semiconductor industry, in catalytic converters, in photography, jewelry manufacture, and in electronics. Accumulation of the element via inhalation can occur in kidney, spleen and liver and its excretion is possible via the urine and biliary tract.

Cadmium: affects the heart, bone and tests. Potential sources of it are drinking water, processed foods, cigarette smoke, paint pigments, silver polish. Smoking and high sugar directly affect the concentration.

Copper: is present in all body tissues but concentrated in liver, brain and hair. Sources of copper are whole grains, nuts, seeds, beans, liver and selfish. Loss of these can lead to anemia, neural degeneration, lung and bone problem.

Zinc: direct sources of zinc are whole grains, nuts, seeds, seafood. Growth and repair of any tissue is dependent on zinc as an activating cofactor for (DNA/RNA) and it is vital to the healing of wounds and skin disorder. Immunological effects of zinc deficiency is similar to those of AIDS.

Molybdenum: is an activator of specific enzymes such as: xanthine oxidase (catalyzes formation of uric acid), sulfite oxidase (catalyzes oxidation of sulfite to sulfate), and aldehyde dehydrogenase (catalyzes oxidation of aldehydes). Possible effects or symptoms consistent with Mo deficiency are: subnormal uric acid in blood and urine, sensitivity or reactivity to sulfites, protein intolerance (specifically to sulfur-bearing amino acids), and sensitivity or reactivity to aldehydes. True Mo deficiency is uncommon but may result from: a poor−quality diet, gastrointestinal dysfunctions, or tungsten exposure. Because normal blood and blood cell Mo levels are very low (a few parts per billion), blood measurement is not an appropriate tissue for confirmation of subnormal molybdenum. Confirmatory tests for Mo deficiency include measurement of urine sulfite concentration (increased in Mo deficiency), measurement of blood/urine uric acid level (decreased in Mo deficiency), and measurement of urinary Mo content.

Toxic metals may exchange between blood plasma and erythrocyte after a person is exposed. Toxic elements are associated with a number of different toxicological effects which begin to manifest when a particular toxic elements is present in the human body above a certain threshold.

In Chapter six, results show that (NAA) of rock samples around the Choke Mountain range gave values of (Al, Na, Cd, Rb, Ge, Ru, Cu, W, Cr, As, Eu, La, Mn, K, Mo, Re, Au, Sr, Ga, Xe, Ga, Te). Some harmful rare earth elements (Rb, Mn, As, Sr, Cr, Cd, Te, Mo, Cu) were observed in the rock samples.

Drawing on the findings of the detailed studies in the communities of the Choke Mountain which is the water tower of the Blue Nile River and its tributaries, it is argued that the instrumental neutron activation analysis approach is an appropriate method both for assessing the impact of the environment and for engaging communities in sustainable adaptation option.

In Chapter seven, results show that (NAA) of rock samples around the Blue Nile Gorge gave values of (Cu, Al, Cl, Pr, Na, In, Rh, Mn, Mg, Ag, Ru, Ce, Yb, W, Er). Among the elements (Ce, Pr, Er, and Yb) are rare earth elements. Heavy metals that most commonly cause problems include (Al, Cu, and Mn).

All heavy metal poisoning and chemical toxicity lead to the accumulation of toxins in our tissues and organs causing nutritional deficiencies, hormonal imbalances and the breakdown of the immune system, the central nervous system, and the organs of the body.

Some of these elements accumulate in the body, so there is no truly safe exposure limit for those elements. As with the other elements, aluminum is observed and accumulated in the body, and has been linked to serious illnesses including osteoporosis, extreme nervousness, anemia, headache, decreased liver and kidney functions, forgetfulness, speech disturbances and memory loss.

It was noticeable that by using the isotropic neutron source, some radioisotopes with great importance in nuclear medicine could be produced (Au^{198}, Mn^{56}, Cu^{64}, Ir^{194}, In^{116}, Zn^{64}, As^{56}, Cd^{117}, Cr^{51}, Al^{28}, W^{187}, Mo^{99}). For harmful rare earth elements include (Ce^{143}, Pr^{142}, Er^{171}, Yb^{177}, Rb^{88}, Mn^{56}, As^{76}, Sr^{87}, Cr^{51}, Cd^{117}, Te^{131}, Mo^{99}, Cu^{64}), for industrial elements include (Au^{198}, Ag^{108}, Cu^{64}, Al^{28}, Pt^{197}, La^{140}, Zn^{64}, Pd^{111}), for Agricultural elements include (K^{42}, Mg^{27}, Ni^{64}, Cu^{64}, Cl^{38}, Mn^{56}, Mo^{99}, Zn^{64}), for Heavy metals include (Mn^{56}, Cu^{64}, Pd^{111}, Ga^{72}, Ta^{182}, Zn^{64}, Er^{171}, Al^{28}, Au^{198}, Ag^{108}, Sc^{46}, Cd^{117}, La^{140}, Mo^{99}).

8.4 Recommendations:

Drawing on the research findings, some suggestions can be made to inform future sustainability practices in the community. The followings are some of these suggestions/recommendations.

- Promotion of awareness about the danger of soil erosion and encourage responsibility
- A good working relations has been established with community leaders and extension officers.
- Support from the community leaders is vital for successful implementations of

the decision support system

- The program will be demonstrated to users and the environmental science department at the University of Debre Markos University, and the university will provide a backup support system.
- A scientific research decision support system will be provided to:
    1. Extension officers at the regional offices of the department of Agriculture, The national department of Agriculture,
    2. The community leaders and small−scale farmers

Therefore, it is recommended that developed countries provide financial and other assistance to enhance the effectiveness of their laboratories and to provide assistance through the IAEA in identifying fields of application where neutron activation analysis can be of enhanced value to developing countries.

8.5 Future Research:

Future research could target beneficiaries of sustainability initiatives specially to build a comprehensive understanding of their sustainability needs and requirements and the scale of the challenges they face. It also be targeted at establishing how sustainability initiatives facilitate capacity building and enhancing resilience in communities. This would help to test and verify the contribution of the systems thinking approach in sustainability of the society.

During the last several years, Ethiopia has been implementing five−year rolling plans with the objective of eradicating poverty. This is due to critical role played by agriculture in achieving sustainable development and poverty reduction which is now being well recognized in the international arena.

The Federal Government of Ethiopia has recently formulated policies that have encouraged investments in the soil mineral and agricultural sectors in order to reduce her overdependence on alternative source of revenues from oil and gas and also household consumptions. Such revenues can only be harnessed after the mineral resources have passed through exploration, mining and processing stages. Each of the stages has both health and environmental implications (Aigbedion & Iyayi, 2007).

The solid mineral deposits that are invested and jostling for in the country have been listed by (Adetunji et al., 2005) to include coal, columbite, cassiterite, marble, limestone, clay, bitumen and tantalite. Others include granite, lead, zinc, gold and barite. The history of mining of some of these minerals dated back to the colonial days and they have generated substantial revenue to the government and contributed immensely to the socioeconomic developments of their hast communities (Adetunji et al., 2005). These desirable efforts are overshadowed by the adverse efforts that have been listed by (Olaleye & Oluyemi, 2010; Aigbedion & Iyayi, 2007; Mokobia et al., 2006; Mokobia & Balogun, 2004) to include ecological disturbances, destruction of natural flora and fauna, pollution of air, land and water, instability of soil and rock masses, land scape degradation and radiation hazards.

Limestone occur extensively in all the sedimentary basin in Ethiopia and are being mined daily for various purposes. Many investors are mindless of the environmental consequences of their mining activities so long as not to disrupt production at their factories. This study will have an attempt to generate information on the elemental composition of some limestone samples that will be useful in planning some remedial measures to cushion some of the effects of mining limestone in Ethiopia.

Also the extreme form of disparity existing in the world in terms of production, consumption and access to agriculture is a major concern. Enhanced development is thus, a necessity to left people out of the agricultural poverty trap and provision of environmentally sound agricultural services, integral to poverty alleviation and sustainable development since it fulfills the basic human needs of nutrition, shelter and clothing, in addition to health and education. This necessitates policy formulations such that the concerns to be addressed encompass the various dimensions of agriculture in terms of accessibility, convenience, affordability, cleanliness, efficiency, current cost, and environmental <spin-off>as well as development. $INAA$ being a multi$-$element analytical procedure reduces the high cost resulting from reactor costs, analytical services and travel. The large number of elements and samples that can be determined simultaneously offsets analytical services if cost is determined on a per analytic bases.

No discernible changes in the sample can generally be observed, however, the sample could be slightly radioactive due to the production of long half$-$life radionuclides.

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Appendix A

List of Publications

- Asres, Y. H, A. K. Chaubey, Awoke, T. H., and Dilbetigle, A. M., (2011). Elemental Analysis Alluvial soil from Blue Nile Basin Using Neutron Activation Analysis, East Gojjam, Ethiopia, E-International Scientific Research Journal Consortium, 3(4), pp232-242

- Awoke T. H., A. K. Chaubey, Asres Y. H. and Dilbetigle A. M. (2012). Application of Instrumental Neutron Activation Analysis of various rocks from areas around Debre Berhan city,Ethiopia,International Journal of Research and Reviews in Applied Science, vol.12, issue 1

- Awoke T. H., A. K. Chaubey, Tilahun Tesfaye, and Asres Y. H. (2012). Application of Instrumental Neutron Activation Analysis Technique for investigation of air pollution in Addis Ababa University, Natural and Computational Science College, Ethiopia, International Journal of Scientific and Engineering Research, vol.3, issue 10

- Asres, Y. H., A. K. Chaubey, Awoke, T. H. and Dilbetigle, A. M.(2013). Instrumental Neutron Activation Analysis of Rock Samples from Blue Nile Gorge, East Gojjam, Ethiopia,International Journal of Scientific and Engineering Research (IJSER), Volume 4, Issue 1

- Asres, Y. H., A. K. Chaubey, Awoke, T. H. and Dilbetigle, A.M, (Submitted), Elemental analysis of soil samples using Neutron Activation Techniques from farmlands of Yebrage Hawariat, East Gojjam, Ethiopia, International Journal of Environmental Science

- Awoke T. H., A. K., Chaubey, Asres Yihunie Hibstie (2013).Investigation of traffic air Pollution in Addis Ababa City around Selected Bus Station using Instrumental Neutron Activation Technique, International Journal of Basic and Applied science Insan Akademika Publications, vol.03 No.01,pp613-620

- Asres, Y. H., A. K. Chaubey, Awoke, T. H. and Dilbetigle, A. M. (2013), Thermal Neutron Activation Technique of Rock Sample From Choke Mountain, East Gojjam, Ethiopia, International Journal of Basic and Applied Science,vol.01, No.04, pp694-704

- M. Dilbetigle Assefa, A. K. Chaubey, Awoke Taddesse and A. Yihunie Hibstie, (2013) Relative determination of Micronutrients of different Species of Teff (Eragretis) Seeds of Ethiopia Origin by calibration Free Laser Induced Breakdown Spectroscopy Techniques, Advanced Journal of Food Science and Technology,vol.5(3):332-338

- M.Dilbetigle Assefa, A. K. Chaubey, Awoke Taddesse and A.Yihunie Hibstie, (Submitted) Analysis of Atmospheric Air Polutants Using Lichens as a Bio-monitor by calibration Free Laser Induced Breakdown Spectroscopy Technique, Applied Physics B Journal

- M. Dilbetigle Assefa, A. K. Chaubey, Awoke Taddesse and A.Yihunie Hibstie, (Submitted) Multi-elemental INAA and CF-LIBS Techniques for analysis of Rocks of Ethiopia Tropical Forest Area of Tepi, Europian Journal of Physics